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INORGANIC COMPOSITE MATERIALS IN JAPAN:
Status and Trends

By

M.J. Koczak, K. Prewo, A. Mortensen, S. Fishman, M. Barsoum, and R. Gottschall

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Japan's high performance composite research activities and applications are reviewed by six authors who teamed as a study group in September 1988 to assess selected research areas of composite activities. This article focuses on metal and ceramic matrix composites and high temperature ceramic reinforcements, excluding carbon fibers and polymer matrix composites. It is the intention of this article to capture the current trends and scientific accomplishments of university, government research laboratories and, to a lesser extent, selected industrial laboratories in these research areas. In addition, an interpretation and forecast of future directions is considered in each topic area based upon current activities and perceived trends.

This publication is approved for official dissemination of technical and scientific information of interest to the Defense research community and the scientific community at large.

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CONTENTS

Cover: Bamboo, a tropical grass, is a natural composite material. Similar to composite structures, it is known for its competitive and prolific growth. The hollow tube and cellular structures provide for excellent structural efficiency at low cost. Based on specific stiffness, bamboo has five times the specific stiffness of steel, e.g., Fe = 59, bamboo = 290. Courtesy of Kohei Aida, from his book on sumie painting titled *Sumie Zen Giho*, 1972.

INTRODUCTION

Japan's high performance composite research activities and applications are reviewed by six authors who teamed as a study group in September 1988 to assess selected research areas of composite activities. These authors and the areas they reviewed are as follows: INTRODUCTION and ADVANCED COMPOSITE MATERIALS PROGRAMS by M.J. Koczak, CERAMIC FIBERS AND WHISKERS by K. Prewo, METAL MATRIX COMPOSITES by A. Mortensen, CERAMIC MATRIX COMPOSITES by M. Barsoum and R. Gottschall, and COMPOSITE APPLICATIONS AND FUTURE DIRECTIONS by M.J. Koczak and S. Fishman. These authors, some of whom were in Japan for the first time during this study visit, were all very favorably impressed by the individuals and facilities that they visited. In general, they found Japanese researchers extremely well informed as to the important issues in composite materials development. The remarkable level of dedication and high technical competence of the engineers and scientists encountered were notable. They were also very thankful of having been very cordially received and for the many in-depth technical discussions. The date of this visit was particularly good because it occurred at a time when substantive discussions were being held in Japan concerning the future directions of composite research. In particular, the establishment of a new Ministry of International Trade and Industry (MITI) sponsored program to address high temperature composites for aerospace applications was under consideration. With a potential starting date of April 1989, this program will mark a major step forward in the goals for Japanese composite researchers. At this period in time, Japan's MITI-sponsored Advanced Composite Materials Program is nearing completion and a review is provided as an introduction of the current research efforts. The subsequent sections shall focus on timely and ongoing specific research in whisker, ceramic fiber, metal, ceramic matrix, and composite application areas. Drs. M. Hyer and A. Dhingra will author a future article focusing on the areas of polymer matrix composites and design aspects of composites in Japan. The study group had selected the aforementioned topics based upon the members' fundamental scientific interest and their areas of current research and expertise. This article focuses on metal and ceramic matrix composites and high temperature ceramic reinforcements, excluding carbon fibers and polymer matrix composites. It is the intention of this article to capture the current trends and scientific accomplishments of university, government research laboratories and, to a lesser extent, selected industrial laboratories in these research areas. In addition, an interpretation and forecast of future directions is considered in each topic area based upon current activities and perceived trends.

The special features that highlight the expeditious Japanese research and applications efforts are: their international perspective, an export-driven economy, and close financial-government and industrial cooperation with a research sector currently driven by commerce rather than defense motivated products. In comparison to the United States, the driving force in Japan is currently the commercial sector (i.e., automotive, information, industrial machines, electronic), and their efforts should be viewed in this context. In contrast, the United States aerospace and defense community provides the initial motivation with commercial areas, e.g., automotive, serving as secondary driving forces. Also, Japan's internal and external market perspective, international materials outlook, and fierce internal competitiveness alter and provide a focus to specific composite material developments. Therefore, the relative strengths of Japan/U.S. research and development areas (e.g., textile/fibers, petroleum, chemical, steel, automotive, aerospace structure, and propulsion industry) must be weighed when a comparison of relative strengths and weaknesses is considered. On this basis, low cost, mass-production applications (e.g., piston inserts made from squeeze-cast metal matrix composites) are sought by the commercial sector in Japan *vis a vis* more costly, higher performance applications associated with the higher temperature, high specific strength, and stiffness requirements essential for the U.S. aerospace industry.

The development of Japan's composite materials technology has been highlighted by a coordinated industry and government research effort, funded by MITI through the Research and Development Institute of Metals and Composites for Future Industries, named the "Jesedai Program." Several developments in high temperature metals and polymer and metal matrix composites have been achieved over the 8-year program. In the composites area, the areas of fiber and whisker developments, ceramic powder processing, and applications of metal matrix composites are particularly notable and will be reviewed. Toyota's application of wear-resistant metal matrix composites for automotive diesel piston fabrication continues to be significant with other structural and wear applications currently being considered and sought. The efforts in ceramic matrix composites are more on a laboratory and developmental stage and have not matured to a manufacturing production stage with the notable exception of the Nissan turbocharger (see the CERAMIC MATRIX COMPOSITES section). Notably, these ceramic matrix composite efforts are more than embryonic, since an accelerated basis of growth can be established from monolithic to reinforced ceramics aided by the world-class efforts in ceramic processing, fiber and whisker development, and modification.

REVIEW OF JAPAN'S
ADVANCED COMPOSITE MATERIALS PROGRAM

BACKGROUND

A salient feature in the understanding of Japan's composite research activities when compared to Europe and the United States is Japan's research and development motivation and market. This will be considered in terms of fiber/whisker development and metal and ceramic matrix composites. Currently, Japanese fiber developments are acknowledged leaders in carbon and silicon carbide fiber as well as whisker modification. In order to produce metal and ceramic matrix composite shapes and components, the fiber and whisker efforts can be combined with "know-how" in the powder processing and monolithic component development and production sectors. One scenario is that the major metal and ceramic companies (Kobe, Nippon Steel, Mitsubishi Metals, Kyocera, Showa Denko, etc.) can use the research and development efforts to produce prototype, semi, and finished products and/or components (e.g., pistons, turbochargers, cutting tools, etc.). A second and more significant version is for complete system applications and redesign (e.g., turbine engine, aircraft, helicopter, and satellite applications). Structural, design analysis, and tradeoff studies are being considered for composite materials substitution for future applications (e.g., aircraft and aerospace structures and propulsion). In the example cited in Figure 1, for the FJR 710 turbine engine application, structural components are being cited in a design trade study with the possible substitution of FRP (e.g., C/PI, C, Kevlar/epoxy), FRM (e.g., SiC/Mg, SiC/Ti, C/Al, and Al_2O_3/Mg), and carbon/carbon for selected component applications. The major manufacturing companies and heavy industries (e.g., Toyota and Nissan Motors, Fuji, Mitsubishi, and Kawasaki Heavy Industries, etc.) must assess the relative cost effectiveness of composites versus current isotropic materials in their current and future markets for vertical integration for future products and market penetration and expansion, i.e., the aerospace market (e.g., turbine engine, helicopter, commercial transport aircraft, satellite launch, and communication systems).

In the commercial and defense areas of polymer and metal matrix composites, the potential markets are being carefully assessed. Japan must consider and combine a technical, financial, and political decision for future market expansion and development. A higher risk and payoff research area involves ceramic matrix composites. The excellent research efforts sponsored by MITI in monolithic ceramic and glass processing can be expanded and combined with a superiority that has been established in carbon, oxide, and carbide fiber and whisker developments by several manufacturers (e.g., Toray, Tonen, Ube, Nippon Carbon, etc.). In a concerted effort, research and industrial application in ceramic matrix composite efforts can be significant. The questions of design, reliability, and market size/growth remain imponderable with varying future market estimates. Nevertheless, considering the Japanese superiority in the areas of cutting tools, ceramic engine components, substrates and electronic packages, industrial dies and bearings, electro-optics, and sensor technology, the development of ceramic matrix composites can be very strong since their presence is well established. The

production basis of ceramic manufacturing processing and reliability have been established in monolithic ceramic structures. For inducement, a viable commercial market must be established as a driving force for continued research in elevated temperature fiber and ceramic matrix composites.

Therefore, the application of ceramic composite structures lies in the cost-effective processing of low cost fiber composites coupled with the uniform processing, inspectability, and structural reliability of the components.

Fan Moving Blade
(FRP: C,G/Epoxy)
(FRM: B/Al)

Fan Frame
(FRP: C/Epoxy)

Entrance Guide Vanes
(FRP: C/Epoxy)

Compressor Rear Moving
and Stator Blades
(FRM: SiC/Al)

Exhaust Frame
(FRS, FRC)

Nacelle
(FRP: C/BMI)
(FRP: C/PI)

Front Frame
(FRP: C,G/PI)
(FRM: Al₂O₃/Mg)

Compressor Front
Moving & Stator Blades
(FRP: C/PI)

High Pressure
Turbine (C/C)

Combustion
Chamber (C/C)
Turbine
Nozzle (FRC)

Low Pressure
Turbine (FRM)

Inner Cowl/Nozzle
(FRP: C/PI)

Tail Cone
(C/C)

Spinner
(FRP: G/Epoxy)

Fan Disk
(FRM: SiC/Ti)

Fan Shaft
(FRM: C/Al)

Compressor
Rear Casing
(FRM: SiC/Ti)

Low Pressure
Turbine Casing
(C/C)

Fan Casing
(FRP: Kevlar/Epoxy)

Compressor
Front Casing
(FRM: SiC/Al)
(FRP: C/PI)

High Pressure
Turbine Casing
(C/C)

Gearbox
(FRP: C/PI)
(FRM: SiC/Mg)

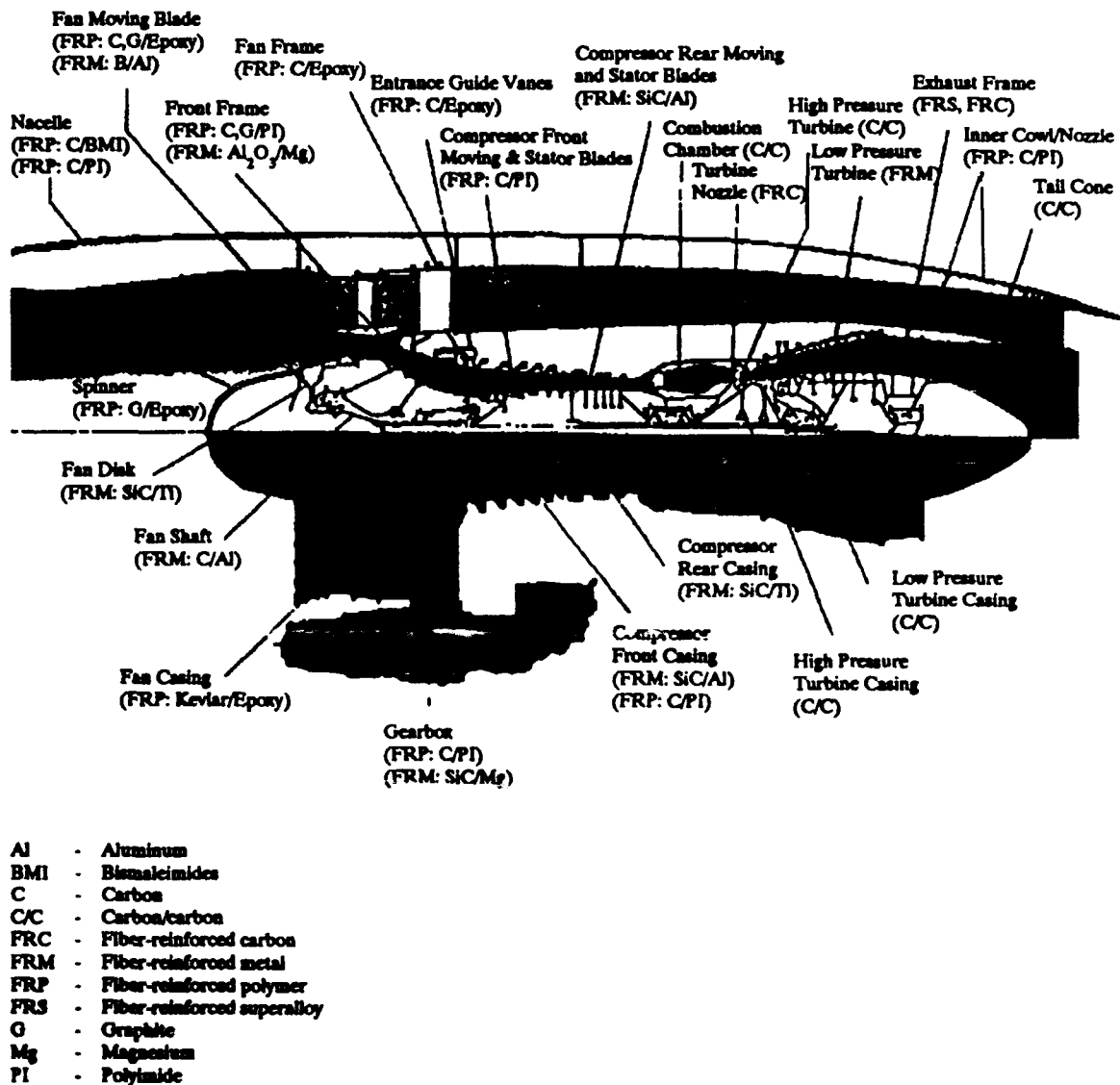| Al	-	Aluminum
BMI	-	Bismaleimides
C	-	Carbon
C/C	-	Carbon/carbon
FRC	-	Fiber-reinforced carbon
FRM	-	Fiber-reinforced metal
FRP	-	Fiber-reinforced polymer
FRS	-	Fiber-reinforced superalloy
G	-	Graphite
Mg	-	Magnesium
PI	-	Polyimide

Figure 1.   The potential applications of composites in a FJR710 gas turbine engine.
Adapted, by permission, from Ref 4.

ADVANCED COMPOSITE MATERIALS PROGRAM

Program Background and Goals

A focus for composite research in Japan has been the Advanced Composite Materials (ACM) Program. The progress of the Research and Development Institute of Metals and Composites for Future Industries, or Jesedai Program, shall be highlighted. It is near completion and can serve as a springboard for the next generation composite materials efforts. The functional organization for the ACM research and development (R&D) team as well as the members' area of expertise (e.g., polymer/metal matrix composites, quality evaluation, and design technology) are provided in Figure 2. The research team is mainly composed of manufacturing companies and fiber companies coupled with government research laboratories and three universities. The ACM program was initiated in 1981, and after an 8-year effort it has recently been concluded. It has repeatedly been reviewed by Hayashi (Ref 1-3) and Minoda (Ref 4,5). In addition, an International Symposium on Basic Technologies for Future Industries--Materials Development and Technology Innovation was held in Kobe, Japan, in March 1988 (Ref 4). The funding was initiated by the Agency of Industrial Science and Technology (AIST) under MITI. These efforts centered upon polymer and metal matrix composites with component goals relating to aerospace, automotive, and engine applications (Figure 3). In metal and polymer matrix composites there have been target goals as a function of temperature that are compared to monolithic unreinforced materials. Tables 1 and 2 provide the property

base that was generated for selected metal (e.g., Al and Ti-6Al-4V) and polyimide matrix composite systems, respectively. The efforts were centered about providing reliable design information and Weibull statistics for confident application. It is informative to compare material costs with fiber and product costs, e.g., yen per gram or dollars per pound for an automobile as detailed in Figure 4. As shown from a cost-performance perspective, the use of glass fiber may be justified for an automotive application; however, aramid, graphite, and whiskers may be prohibitively expensive for commercial applications except for limited, niche production applications.

Polymer Matrix Composites

In the area of polymer matrix composites, the ACM research involved development of heat-resistant matrix systems (e.g., epoxy, polyimide, and polyphenylquinoxarine (PPQ) and their composites). Three types of epoxies were cited: a highly cross-linked system, a heat-resistant system, and a hydrophobic system. For polyimide systems, a series of three modifications was considered: (1) terminal group selection (TPI), (2) main chain modification (MPI), and (3) oligomer structure modification (OPI); the target properties and experimental results are tabulated in Table 3 for graphite composite structures

In an investigation of fabrication technology, three-dimensional (3-D) woven preforms and composites were evaluated. The properties of the 3-D compared favorably with the strength and modulus of a crossply laminate system with a significant increase in the interlaminar shear strength (Table 4). The manufacturing topics involved:
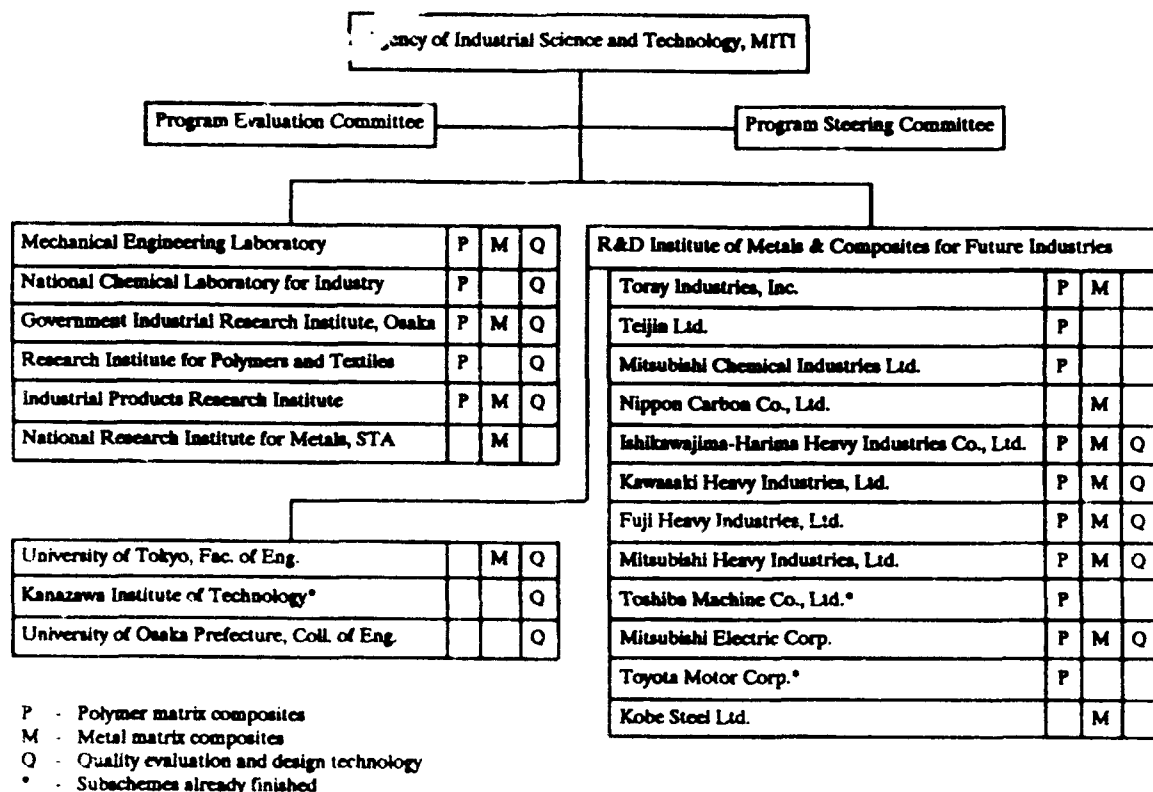
Agency of Industrial Science and Technology, MITI

Program Evaluation Committee	Program Steering Committee

Mechanical Engineering Laboratory	P	M	Q
National Chemical Laboratory for Industry	P		Q
Government Industrial Research Institute, Osaka	P	M	Q
Research Institute for Polymers and Textiles	P		Q
Industrial Products Research Institute	P	M	Q
National Research Institute for Metals, STA		M	

University of Tokyo, Fac. of Eng.		M	Q
Kanazawa Institute of Technology*			Q
University of Osaka Prefecture, Coll. of Eng.			Q

R&D Institute of Metals & Composites for Future Industries			
Toray Industries, Inc.	P	M	
Teijin Ltd.	P		
Mitsubishi Chemical Industries Ltd.	P		
Nippon Carbon Co., Ltd.		M	
Ishikawajima-Harima Heavy Industries Co., Ltd.	P	M	Q
Kawasaki Heavy Industries, Ltd.	P	M	Q
Fuji Heavy Industries, Ltd.	P	M	Q
Mitsubishi Heavy Industries, Ltd.	P	M	Q
Toshiba Machine Co., Ltd.*	P		
Mitsubishi Electric Corp.	P	M	Q
Toyota Motor Corp.*	P		
Kobe Steel Ltd.		M	

P - Polymer matrix composites
M - Metal matrix composites
Q - Quality evaluation and design technology
* - Subschemes already finished

Figure 2. Organization of the Advanced Composite Materials (ACM) Program detailing areas of research. Courtesy of Y. Minoda.

Research Topics	Industry
Easily Processible Heat Resistant Polymers, Curing Process Control Systems	Kawasaki Heavy Ind.
Composite Tape Laying Machine	Toshiba Machine Co.
Carbon Fiber/Polyimide Integrated Box Structures	Fuji Heavy Ind.
Continuous Bend Forming Processes	Mitsubishi Heavy Ind.
Graphite Epoxy Composite Tube Structures for Large Space Structures	Mitsubishi Electric Corp.
Temperature Resin Injection Molding	Toyota Motor Corp.
Prestretched Press Forming	Fuji Heavy Ind.
Microwave Curing	Fuji Heavy Ind.
Hot Hydraulic Forming	Kawasaki Heavy Ind.

Metal Matrix Composites

The study of metal matrix composites involved two major areas: high performance prepregs and wires/sheets and fabrication technology. In addition, design technology was considered. Target properties were cited for materials and structural indices with regard to specific strength and specific modulus at ambient and elevated temperatures (Tables 5 and 6). The target properties are for tension and compression applications in aircraft structures and propulsion systems as well as space structures. Figures 5 and 6 reflect the static specific properties with regard to these program target goals

for carbon (CF), SiC, and boron filaments in aluminum and titanium matrix systems. The specific properties of several aluminum and titanium-based metal matrix systems with graphite and SiC [e.g., Avco (CVD) filaments and Nicalon (PCS) fibers] are compared with varying processing routes [e.g., hot isostatic pressing (HIP), extrusion hot pressing, etc. (Figure 7)]. The elevated temperature specific strength response up to 700 K is also compared for several aluminum and titanium matrix systems versus isotropic titanium, nickel, and aluminum matrix systems (Figure 8). In general, the highest specific properties were achieved with M40 graphite/6061 (V_f = 0.67) aluminum at ambient temperatures and Avco SiC filaments in a Ti-6Al-4V matrix maintaining stable strength levels at elevated temperatures. Elevated temperature studies of titanium alloys with Avco SiC filaments (e.g., SCS-2, SCS-6) revealed that Ti-15Mo-5Zr-3Al provided for ambient strengths of 2,000 MPa and strengths at 450 °C of nearly 1,700 MPa with a 38-percent volume fraction.
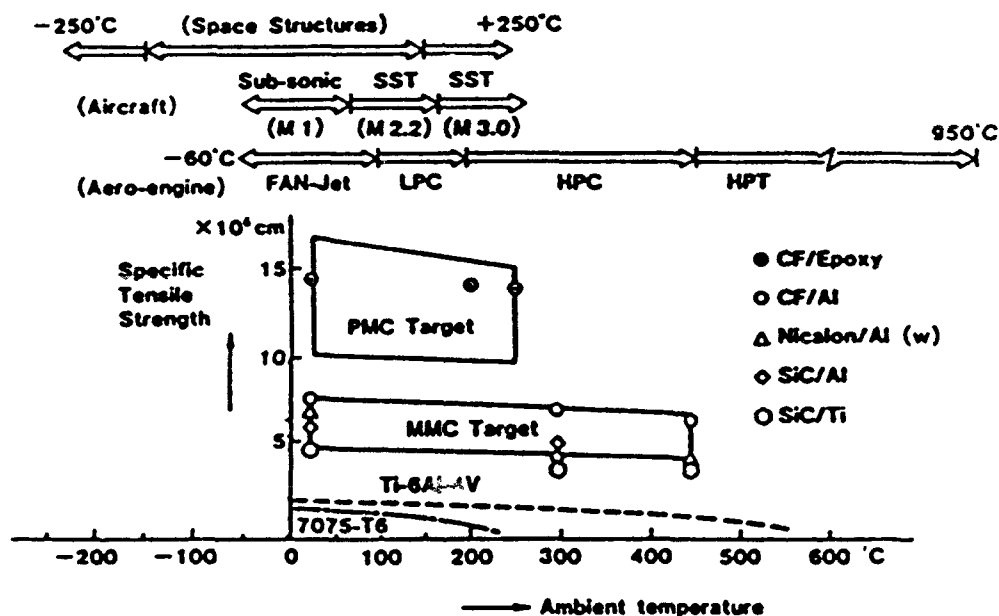

Figure 3. Target goals and properties for metal matrix (MMC) and polymer matrix composites (PMC). Courtesy of Y. Minoda.

Table 1. Reliability Analysis of UTS of Unidirectional MMC Composites

Item	SiC(Nicalon)/ Al-5.7Ni) Wire (V_f-50%)	SCS-6/Ti-6Al-4V Sheet (V_f-49%)	Target
Year tested	1986	1987	1981
Room Temperature			
No. of data	30	6	
Mean (MPa)	1691	2080	
Standard deviation (MPa)	87.83	120	
Coefficient of variation (%)	5.19	5.7	
Shape parameter	21.32	15.4	
Scale parameter	1732	2150	
B-allowable			
Weibull distribution	1522.46	1747.62	1470
Normal distribution	1550.36	1782.38	
450 °C			
No. of data	27	20	
Mean (MPa)	1689.22	1746	
Standard deviation (MPa)	103.84	44	
Coefficient of variation (%)	6.15	2.5	
Shape parameter	17.71	42.6	
Scale parameter	1737	1767	
B-allowable			
Weibull distribution	1479.54	1655.70	1320
Normal distribution	1512.91	1670.16	

Table 2. Reliability Analysis of UTS of Polyimide
Unidirectional PMC Composites

| Item | TORAYCA T400/ SEPI[a] (MW-1500) | | Target |
	V_f-65%	V_f-70%	
Year tested	1986	1987	1981
Room Temperature			
No. of data	16	30	
Mean (MPa)	2464.75	2582.73	
Standard deviation (MPa)	96.85	110.78	
Coefficient of variation (%)	3.93	4.29	
Shape parameter	26.60	25.70	
Scale parameter	2511.13	2634.85	
B-allowable			
Weibull distribution	2276.74	2360.36	2350
Normal distribution	2291.17	2401.52	
250 °C			
No. of data	9	8	
Mean (MPa)	2326.33	2576.25	
Standard deviation (MPa)	73.92	73.67	
Coefficient of variation (%)	3.18	2.86	
Shape parameter	30.07	33.20	
Scale parameter	2363.50	2613.16	
B-allowable			
Weibull distribution	2148.21	2407.69	2115
Normal distribution	2162.82	2414.38	

[a]SEPI: Soluble end-capped polyimide.

FIBER MATERIAL COST

	$3/lb ¥1/g	$30/lb ¥10/g	$300/lb ¥100/g	$3,000/lb ¥1,000/g	$30,000/lb ¥10,000/g
Fibers	Potassium Titanate Fiber				
	Glass Fiber 0.3 — 0.5	2 — 4			
		Aramid Fiber 5 — 8			
			Si_3N_4 Fiber 200 — 300		
			Boron Fiber 180 — 300		
		Carbon Fiber (PAN) 10 — 50			
			Al_2O_3 Fiber 80 — 120		
	Al_2O_3-SiO_2 Short Fiber 1 — 3	Al_2O_3 Short Fiber 7 — 10			
			SiC Whisker 50 — 120		
Other Materials	Al 0.3 — 0.5	Ti 1.5	Shape Memory Alloy 15 — 30	Ge 250 — 270	GaAs Single Crystal 6,000 — 7,000
	Cu 0.3 — 0.4	Si_3N_4 2.5 — 10			
	Fe 0.07	Engineering Plastics 0.7 — 1.3	ZrO_2 PSZ 15 — 20	Si Wafer 150	Pt 2,500
	Plastics 0.3 — 0.7				
Products	2,000-cc Automobile 1.4 — 1.7				

Figure 4. Relative fiber and material cost compared to the cost of a 2,000-cc automobile. "FRM application for automotive parts," by K. Funatani, *Journal of the Japan Society of Mechanical Engineers* 89(808), 241 (1986). Reprinted by permission of the Japan Society of Mechanical Engineers.

Table 3. Properties of Polyimide Composites[a]

Property	Temperature	TPI	MPI	OPI	Target for First Stage
Tensile strength (GPa)	RT	2.19	1.98	2.33	2.06
	250 °C, 10 min	1.94	1.82	2.18	1.86
	-60 °C, 10 min	1.84	2.18	2.18	1.67
Tensile modulus (GPa)	RT	164	134	153	152
Elongation (%)	RT	1.34	1.38	1.59	1.35
Flexural strength (GPa)	RT	2.47	2.40	2.38	2.01
	250 °C, 10 min	1.82	1.70	1.65	1.53
	250 °C, 500 h	2.43	1.69	1.34	1.32
ILSS[b] (GPa)	RT	0.113	0.067	0.162	0.0882
V_f[c] (%)	--	69.4	64.4	65.0	65

[a]TORAYCA 400/polyimide.

[b]ILSS: Interlaminar shear strength.

[c]V_f: Fiber volume fraction (1 GPa = 102 kgf mm^2).

Processing studies in metal matrix composites included: roll diffusion bonding (Fuji Heavy Industries), fabrication of metal matrix composites by hot isostatic pressing (Kawasaki Heavy Industries, Kobe), and fabrication of SiC whisker/Al squeeze casting and extrusion (Mitsubishi Heavy Industries). Material development studies included heat-resistant matrix systems, aluminum matrix composites with SiC-coated graphite fibers, optimization of SiC filament/Ti matrix composites investigating interfacial reactions, and aluminum metal matrix composites developed from "TORAYCA" and Nicalon SiC/Al composite wires. Evaluation and characterization efforts included fracture mechanics, acoustic emission, delamination crack detection, and nondestructive evaluation approach using millimeter waves for fiber orientation evaluations. A listing of U.S. patents, a series of computer codes, and material property data bases have been generated. Patents include U.S. Patents #4,510,103, #4,670,536, and #4,551,508 in polymer matrix composites; U.S. Patent #4,649,060 for the production of reinforced tapes, wires, and tape for reinforced metals; and U.S. Patent #4,673,541 for the production of carbon fiber-reinforced continuous plastic tubes. Further details can be obtained from:

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In addition, the program was summarized at the International Symposium on Basic Technologies for Future Industries, 22-25 March 1988, in Kobe, Japan.

Table 4. Mechanical Properties of 3-D Fabric Composites
(Uninterlaced Structure, Epoxy/Graphite)

Property	3-D-1	3-D-2	LS[a]
No. of filament (kf)	3	6	3
Content (V_f %)	52.6	50.4	57.1
Specific gravity	1.51	1.49	1.53
Tensile			
Strength (MPa)	--	746	--
Modulus (GPa)	61.8	56.1	62.9
Specific modulus (GPa)	40.9	37.7	41.1
Flexure			
Strength (MPa)	760	673	736
Specific strength (MPa)	503	452	481
Modulus (GPa)	55.3	51.1	55.7
Specific modulus (GPa)	36.6	34.3	36.4
Compressive			
Strength (MPa)	477	415	459
Specific strength (MPa)	316	272	300
Modulus (GPa)	61.4	48.5	62.7
Specific modulus (GPa)	40.7	32.6	41.6
Shearing strength (MPa)	>163	>161	42.1
Poisson's ratio		0.13	

[a]LS: Laminate system.

For Aircraft Structure and Aero-Engine:

P_1 = Target (F/g, E/g) for tensile load
  F_L = 1.52 GPa (155 kgf/mm^2)
  E_L = 100 GPa (1.02 x 10^4 kgf/mm^2)

B_1 = Target (E/g) for buckling load of
  column: compression
  plate: compression, shear
  cylinder: compression, bending,
             torsion, external pressure

E_L = 58.8 GPa (6 x 10^4 kgf/mm^2)

For Space Structure:

P_2 = Target (F/g, E/g) for tensile load
  F_L = 1.03 GPa (105 kgf/mm^2)
  E_L = 152 GPa (1.55 x 10^4 kgf/mm^2)

B_2 = Target (E/g) for buckling load of
  cylinder: compression, bending,
            torsion, external pressure
  E_L = 142 GPa (1.45 x 10^4 kgf/mm^2)

B_3 = Target (E/g) for buckling load of
  column: compression
  plate: compression, shear
  E_L = 316 GPa (3.22 x 10^4 kgf/mm^2)

Table 6. Target of Static Specific Strength and Specific Modulus of FRM at Room Temperature (see Figure 5)

For Aircraft Structure and Aero-Engine:

P_1 = Target (F/g, E/g) for tensile load
  F_L = 2.30 GPa (235 kgf/mm^2)
  E_L = 98.0 GPa (100 x 10^4 kgf/mm^2)

B_1 = Target (E/g) for buckling load of
  column: compression
  plate: compression, shear
  cylinder: compression, bending,
             torsion, external pressure

E_L = 81.3 GPa (0.83 x 10^4 kgf/mm^2)

For Space Structure:

P_2 = Target (F/g, E/g) for tensile load
  F_L = 1.18 GPa (121 kgf/mm^2)
  E_L = 131 GPa (1.34 x 10^4 kgf/mm^2)

B_2 = Target (E/g) for buckling load of
  cylinder: compression, bending,
            torsion, external pressure
  E_L = 139 GPa (1.42 x 10^4 kgf/mm^2)

B_3 = Target (E/g) for buckling load of
  column: compression
  plate: compression, shear
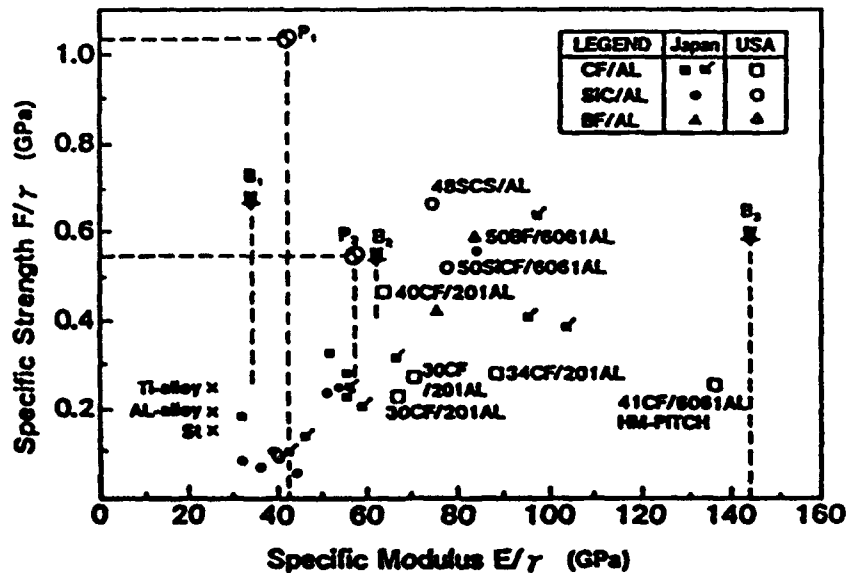 E_L = 324 GPa (3.31 x 10^4 kgf/mm^2)

Figure 5. Static specific strength versus specific modulus of metal matrix composites (MMC) at room temperature. Reprinted, by permission, from Ref 4.
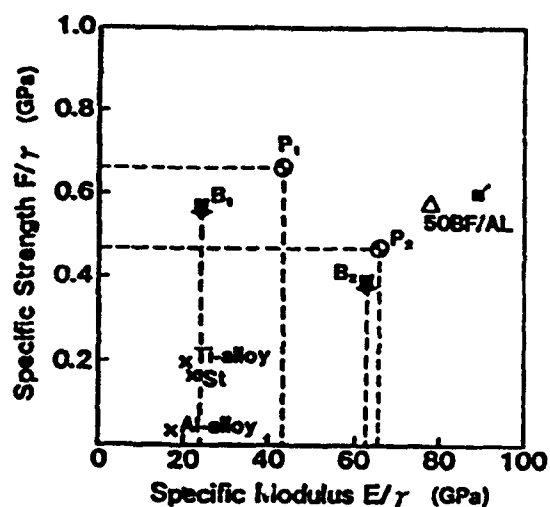
Figure 6. Static specific strength versus specific modulus of metal matrix composites (MMC) at elevated temperature, T = 250 °C. Reprinted, by permission, from Ref 4.
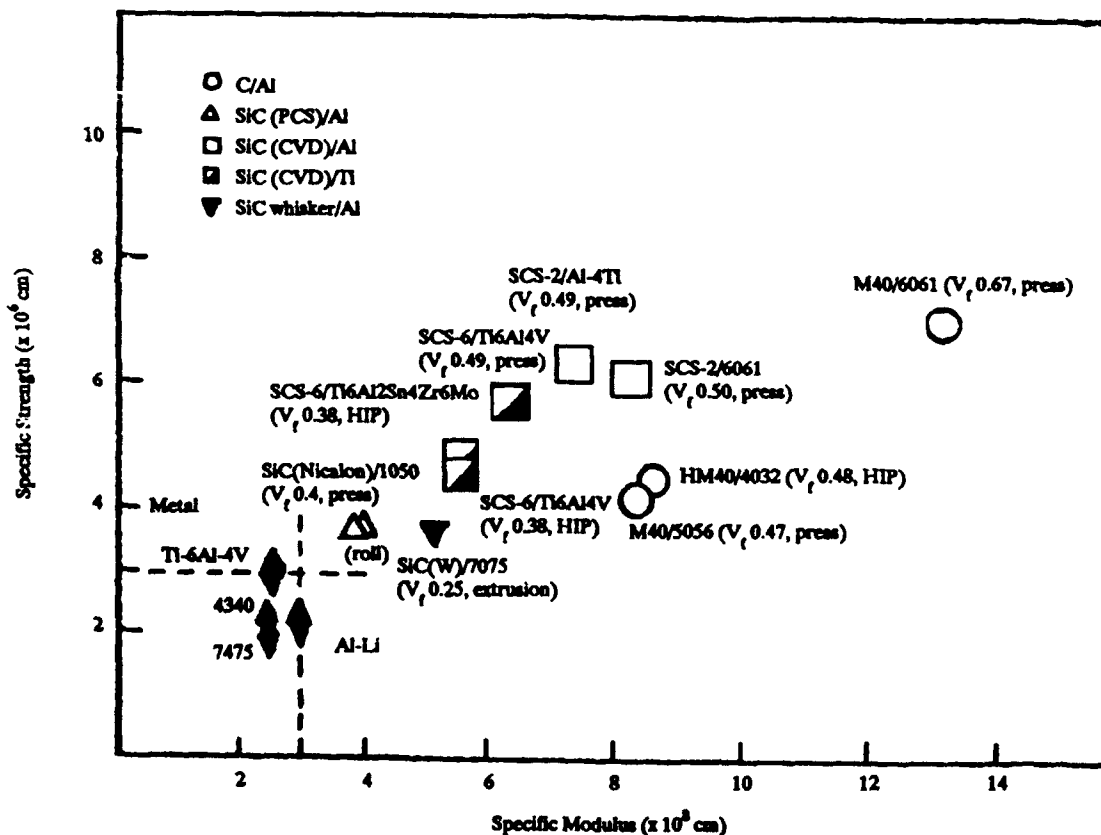

Figure 7. Static specific strength versus specific modulus of aluminum and titanium base metal matrix composites (MMC) at room temperature. Redrawn, by permission, from Ref 5.
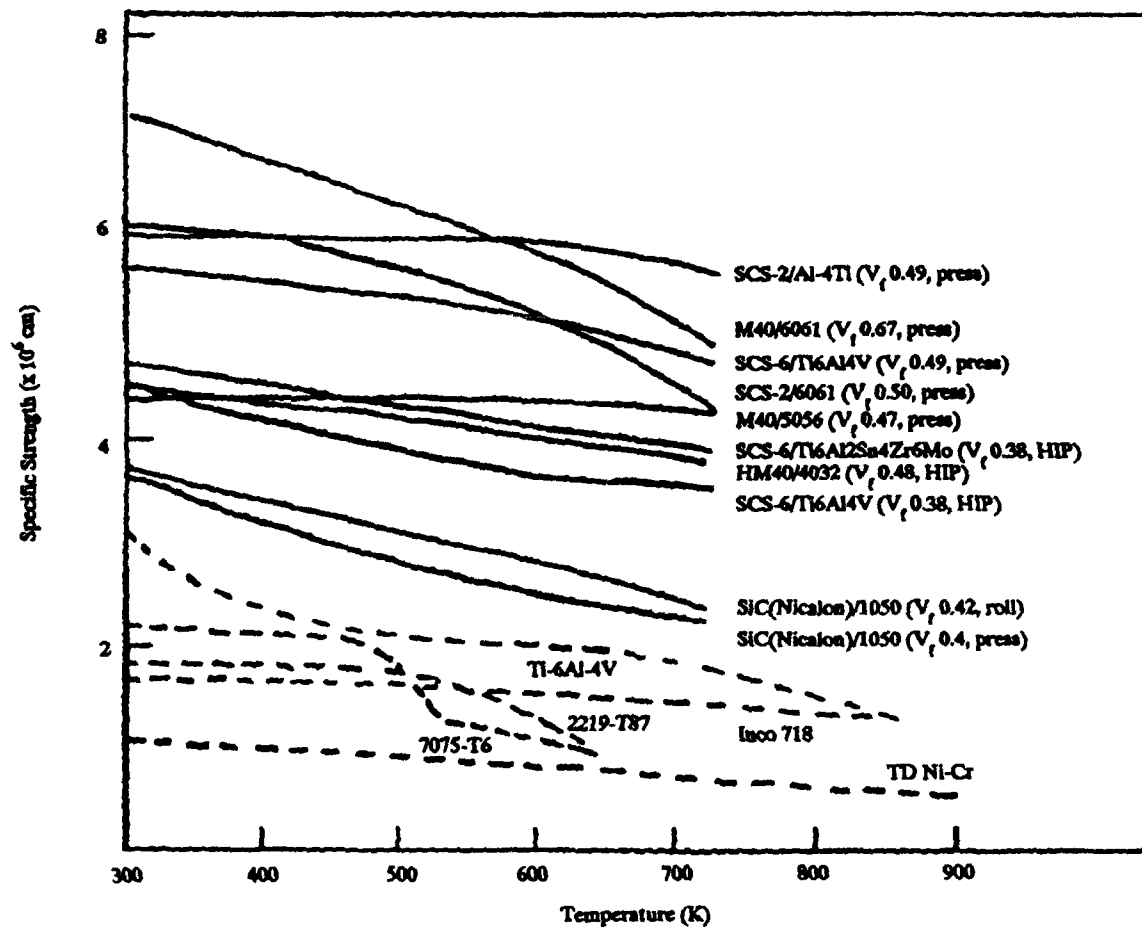
Figure 8. Specific strength of aluminum and titanium base metal matrix composites (MMC) versus temperature. Redrawn, by permission, from Ref 5.

ADVANCED CERAMIC FIBERS

INTRODUCTION

The expansion in focus of Japanese composite materials interests into high temperature regions above 450 °C is spurring a significant revitalization in ceramic fiber technology. The driving force behind this interest stems from three important areas. First is a strong desire on the part of Japanese companies to diversify. Companies traditionally in petroleum, gas, chemical, and heavy industry all want to produce new high performance materials. Second is a government-encouraged and sponsored interest in new applications involving high temperature gas turbines, supersonic vehicles, and energy conversion systems. The MITI composite materials program illustrates this. Third is a realization that these ceramic fibers can be tailored in their characteristics to meet a broad range of performance goals for polymer matrix (PMC), metal matrix (MMC), intermetallic matrix (IMC), and ceramic matrix (CMC) composite applications. It should be noted that the performance goals being set in Japan are of direct relevance to those set in the United States for advanced MMC, IMC, and CMC research initiatives.

A current Japanese leadership position in polymer-derived ceramic fibers appears to be increasing and forms the basis for exploration of high temperature fibers and ceramic chemistries with exceptional potential. Performance goals including stability to 1,800 °C are being contemplated. The availability of lower cost whiskers is being expanded and the issue of health hazard is being overcome through the redirection of fabrication procedures to produce whiskers with diameters greater than 2 microns.

BACKGROUND

There currently exists a broad range of ceramic fibers in Japan. The fibers are in two principal forms: multifilament yarns and whiskers. These two forms have been emphasized due to their potential low cost and suitability for large volume production. Large diameter monofilaments made by chemical vapor deposition (CVD) have received little emphasis presumably because of their higher cost.

It is important to note that the basic technology to develop high performance ceramic fibers has been in existence in Japan since at least the mid-1970s. Notable in the early development stages was the revolutionary work of Professor Yajima and his group at Tohoku University. This work encompassed not only concepts for fiber development but also the use of this chemistry to create monolithic ceramics and binders, and it also included compatibility studies with a wide range of metals including Al, Ti, Fe, Mo, and Cr. Advanced chemistries, including third element additions to enhance high temperature stability, were all part of this early Japanese technology development. Early commercialization of this technology as Nicalon SiC fiber by the Nippon Carbon Co. in about 1976 sparked the interest of researchers around the world in the development of new families of oxidatively stable ceramic matrix composites. Similarly, an aluminum oxide fiber, containing SiO_2, was available from Sumitomo Chemicals with a tensile strength of 2,450 MPa and elastic modulus of 290 GPa.

From its commercialization until the present the Nicalon fiber has been used in Europe and the United States to develop high performance ceramic matrix and metal matrix composites. In contrast, very little interest in this fiber and these systems had been evident in Japan. The difference can be traced to the U.S./European interest in high performance aerospace needs and the contrasting Japanese interest in high volume, lower cost commercial applications. As a result, in Japan, Toyota Motor Co. achieved its major breakthrough in metal matrix composites by commercializing pressure-cast pistons using a lower cost discontinuous oxide fiber. Also, research in advanced composites emphasized the use of whiskers having potential for low cost.

CURRENT STATUS

Japanese companies remain the world leaders in the development of polymer-derived ceramic fibers. A partial list is presented in Table 7. At this time two major companies are producing commercially available "SiC type" fibers (i.e., Nippon Carbon Co. Ltd. and Ube Industries Ltd.). Both have chosen to distribute their fibers in the United States through U.S. companies who have the ability to market fibers to meet Department of Defense contractor requirements. These U.S. intermediaries also have strong interest in the development and sale of their own fiber products. Avco* (for Ube) and Dow Corning (for Nippon Carbon) have thus provided valuable service and also gained firsthand market knowledge. Avco has taken a particularly active role by also marketing Sumitomo's aluminum oxide fiber and Tokai whiskers.

Table 7. Manufacturers' Reported Data for Fibers

Fiber	Manufacturer	Diameter (μ)	Density (g/cm^3)	Elongation Modulus (GPa)	UTS (MPa)
Si-C-O	Nippon Carbon	15	2.55	200	2740
Si-C-Ti-O	Ube	8-10	2.3-2.5	200	2740
Si-N-O	Tonen	10	2.5	300	2500
Si-N-O	Tohoku University	12	2.6	140	1800
Si-O-N	Shimadzu	7-20	3.03	195	4800
Al$_2$O$_3$-15%SiO$_2$	Sumitomo	10-15	3.25	210	1800
99.5% Al$_2$O$_3$	Mitsui Mining	10-13	--	342	1950

*Currently Textron.

A significant upsurge of interest in developing a broader range of ceramic fibers is presently apparent in Japan. Both Nippon Carbon and Ube have made major progress in providing varieties of their fibers to meet specific needs. Both offer fiber variations with tailored electrical properties and Ube also offers fibers with ceramic whiskers or particles attached to their surfaces. The latter hybrid concept, originally developed at Toyota Motor Company Central R&D Labs, is designed to improve pressure-cast metal matrix composite microstructures. Despite the rather small constant demand in the past for these fibers, both companies appear committed to an assured supply to their customers.

Additional concepts for polymer-derived fibers have already been developed in Japan. Notable among these have been two different approaches to creating silicon nitride type fibers. Professor Okamura at the Oarai Branch, Institute for Materials Research of Tohoku University, has developed a fiber that he has shown to possess a tensile strength and elastic modulus of 1,800 MPa and 139 GPa, respectively. While only prepared in laboratory scale quantities, notable evidence exists for strength retention at up to 1,200 °C. By totally separate approaches, workers at the Corporate R&D Labs of Tonen (Toa Nenryo Kogyo K.K.) have developed a Si-N type fiber with a reported strength of 2,500 MPa and an elastic modulus of 300 GPa. This fiber is well on the way to pilot scale production and is awaited by many researchers for potential use at up to 1,200 °C. Another approach to Si-N type fiber production has been announced by Shimadzu Corp. and has resulted in a fiber that the company claims to have a tensile strength and elastic modulus of 4,800 MPa and 195 GPa, respectively. Of interest is the absence of a Japanese producer of a large diameter monofilament.

Neither boron or SiC type fibers are being produced commercially. However, it should be noted that Japanese researchers have been very successful in making high performance composites (particularly titanium matrix composites) using Avco-supplied SiC monofilament and this success should spur additional interest for a domestic supply.

Whisker producers have notably taken action to overcome the potential health hazards of their products. To this end recent emphasis has been placed on production whiskers with diameters of 2 microns or greater. It is still presumed that whisker costs will be below those of continuous fibers, whisker properties can be exceptionally high, and whisker chemistries differ markedly from their polymer-derived counterparts.

Finally, it is also important to remark that Japanese fiber producers are taking steps to move up the "value added" scale toward composite production. Several producers offer their fibers as metal-coated tow, i.e., Nippon Carbon's Al-coated Nicalon and Toray's Al-coated carbon fibers. In a further extension, those companies using polymer precursor routes to fiber production have recognized the potential to also create coatings and ceramic matrices by similar techniques.

FUTURE DIRECTIONS

The recent emphasis by MITI and other Japanese programs on high temperature performance goals has had a profound effect on revitalizing interest in ceramic fibers. Most of the activity appears to be in the polymer-derived compositions and future developments in this area appear to center on the following activities: understanding high temperature performance, developing new processing approaches, and creating new compositions.

High Temperature Stability

The Nicalon and Tyranno fibers are being used to develop an understanding of the stability and resistance to degradation of fibers at elevated temperature. In particular, the groups at Tohoku University (Oarai Branch), Nippon Carbon, and Ube have emphasized this and are working to define the paths to higher temperature performance. Figures 9 and 10 (Ref 6) relate the crystallite size of Nicalon fibers to various heat treatment conditions. It is shown that fiber strength decreases with increasing crystallite size; however, the authors state that the crystallite size effect "is only superficial and what really caused the decline are the pores and faults" that are created during crystallite growth. The work by Dr. Yamamura and his colleagues at Ube has been based on the Tyranno fiber, which contains about 3 percent by weight Ti and has been shown to be more resistant to degradation by thermal exposure (Figure 11) (Ref 7) despite the fact that it contains a very high percentage of oxygen (17.7 percent by weight). The role of the Ti to stabilize fiber performance is serving as a model for researchers in Japan who are now considering the potential for other elemental additions as well. Performance goals at temperatures of 1,700 °C and above are being put forward by MITI and it can be expected that important knowledge as to the mechanisms controlling polymer-derived ceramic performance will be gained.
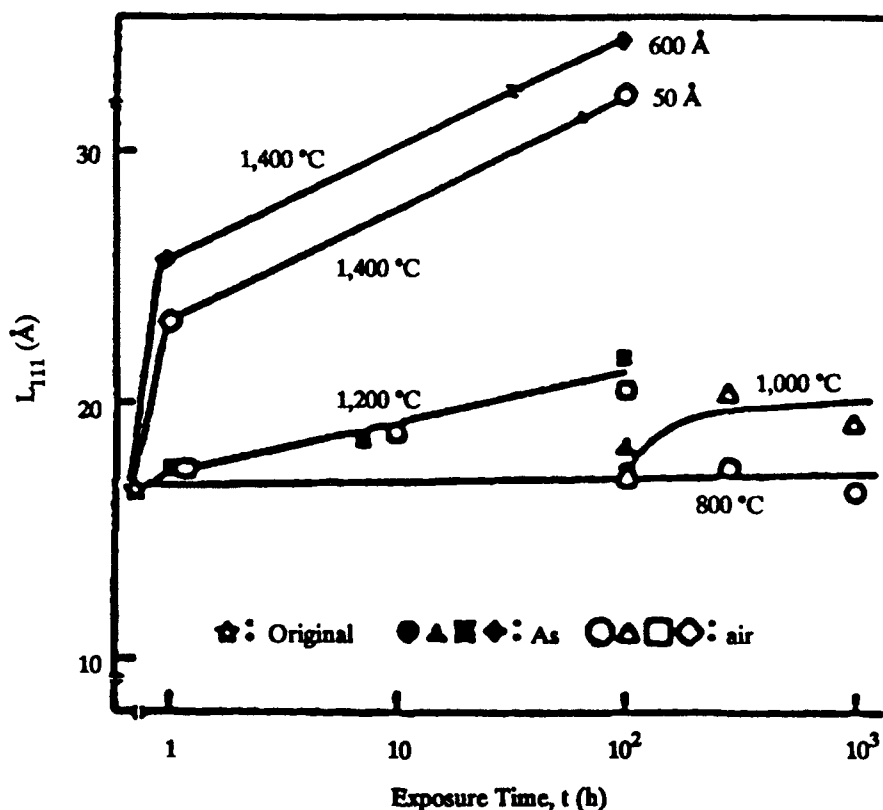

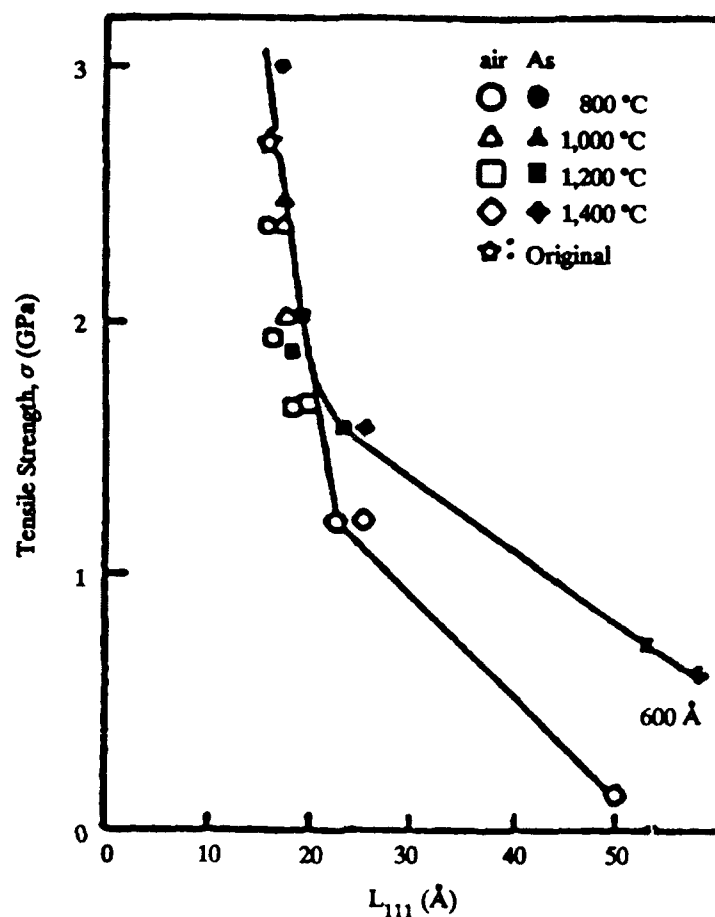Figure 9. Crystallite size β-SiC(L_{111}) after exposure (Ref 6).

Figure 10. Relation between tensile strength of SiC fiber and crystallite size of β-SiC(L_{111}) (Ref 6).

New Processing Approaches

Professor Okamura of Tohoku University (Oarai Branch), working in conjunction with other Japanese researchers and companies, has been exploring alternative processing approaches to create high strength fibers. The general aspects of fiber processing are illustrated in Figure 12 (Ref 8). One of the key steps in current fiber production involves curing in an oxidizing atmosphere. It is in this step that additional oxygen is incorporated into the fiber structure with

the later enhanced creation of CO during high temperature exposure that accompanies grain growth and strength loss (Figure 13) (Ref 8). To eliminate this additional oxygen, a cross-linking step using electron irradiation has been substituted for the oxygen cure. Irradiation in He gas and subsequent heat treatment at 1,200 to 1,600 °C in argon produced fibers that were much more resistant to thermal degradation (Figure 14). The irradiated fibers contained only 3 to 4 percent oxygen by weight while the oxygen-cured fibers contained 10 percent.
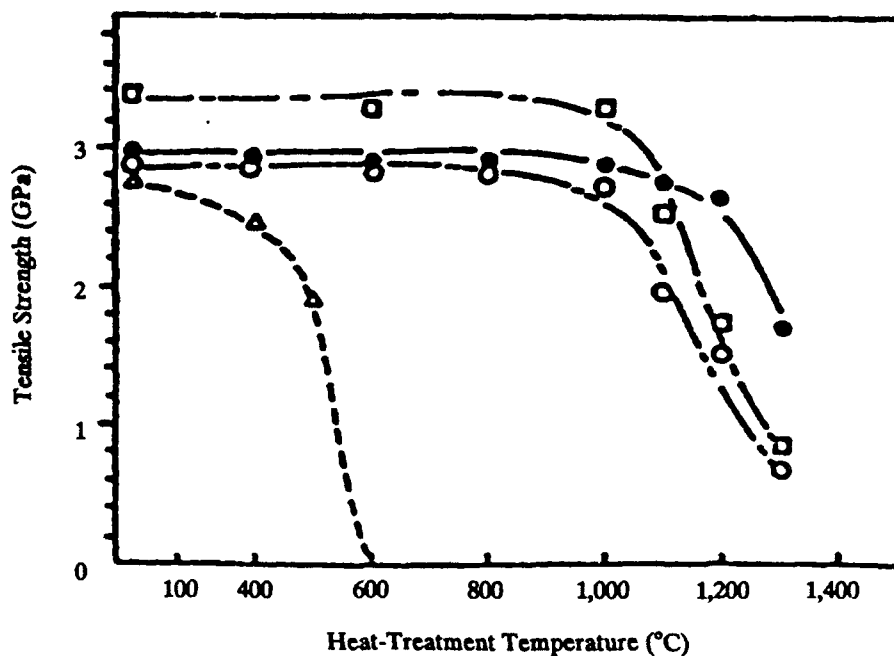
Figure 11. Heat resistance of carbon fiber (Δ), SiC fiber (o), SiC/C fiber (□), and Si-Ti-C-O fiber (·) (1300) (Ref 7). Reprinted with permission from *J. of Mat. Sci.* 23, 2589, T. Yamamura et al. Copyright 1988, Chapman & Hall.
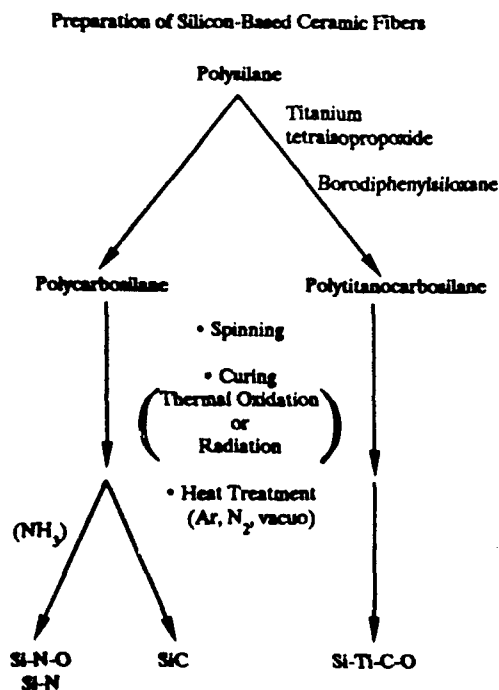

Figure 12. Processing routes for silicon-based ceramic fibers. "Silicon-based ceramic fibers," by K. Okamura et al., *Ceramic Eng. Sci. Proc.* 9(7-8), 909-918 (1988). Reprinted by permission of the American Ceramic Society.
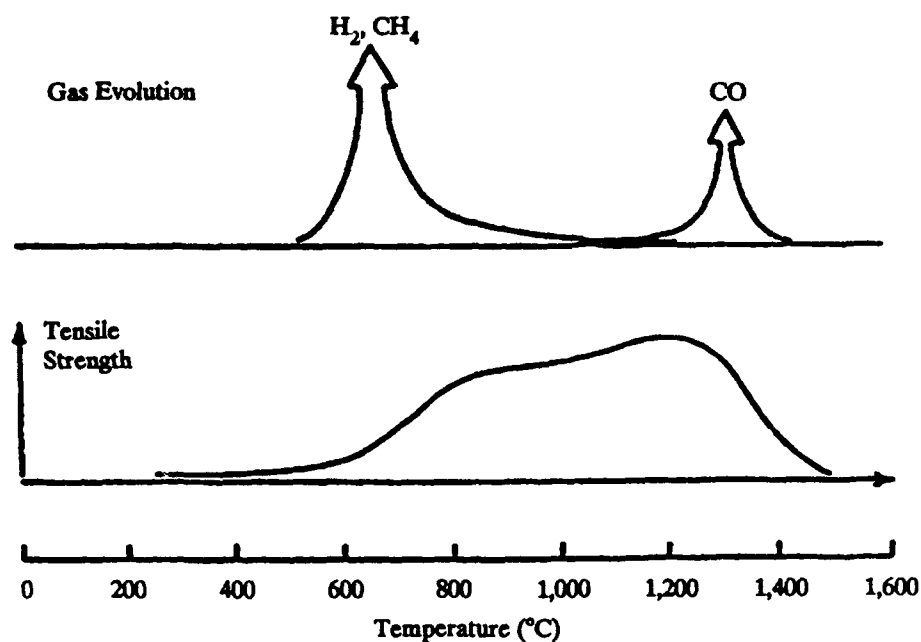
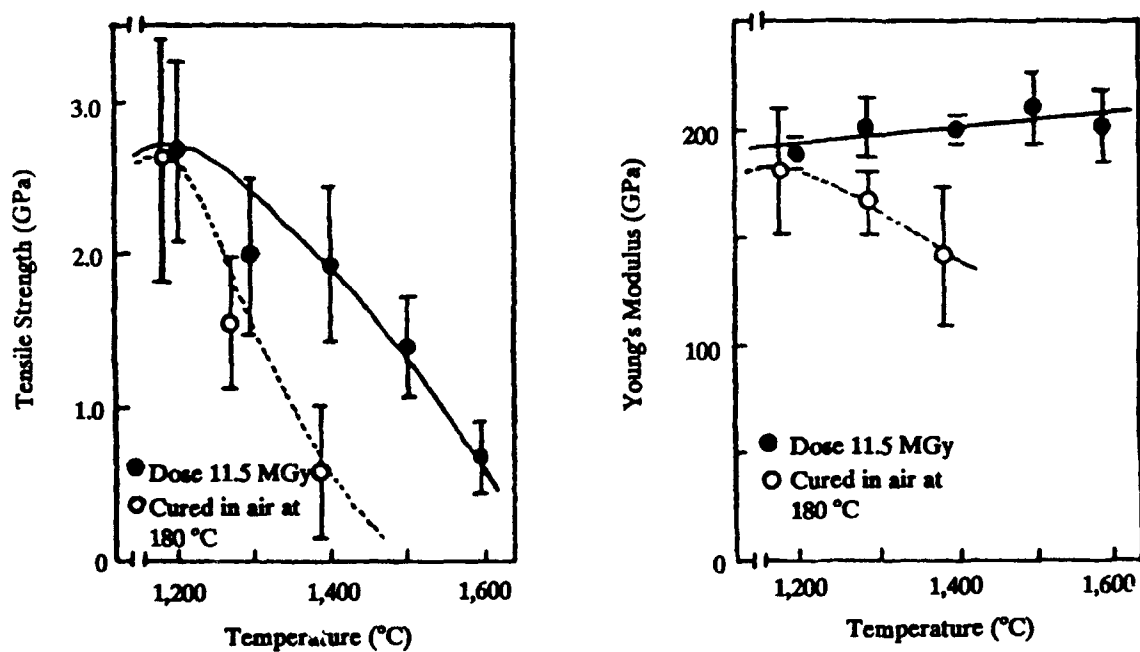Figure 13. Gas evolution from "SiC type" fibers.

Figure 14. Properties of "SiC type" fibers, cured in air or electron irradiation, as a function of heat treatment.

Concepts are also being developed for further increasing fiber properties subsequent to production by high temperature pyrolysis. One such approach is to irradiate the fibers with fast neutrons. By this technique it has been possible to increase fiber tensile strength by increasing fiber density (Figure 15) (Ref 9); unlike in high temperature heat treatment, high densities are achievable presumably due to removal of microporosity.
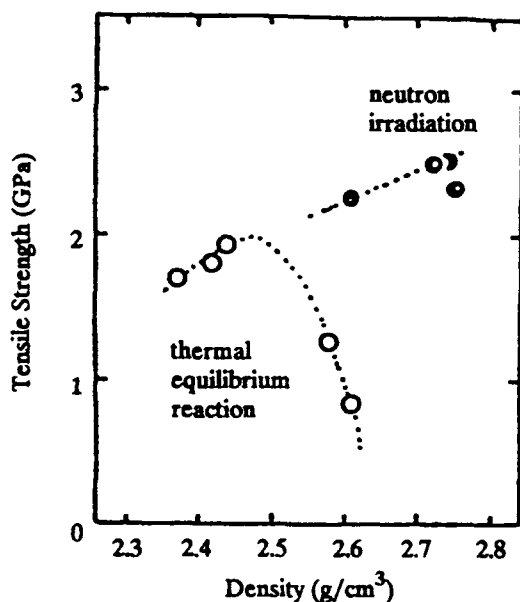

Figure 15. Relationship between tensile strength and density of SiC fibers. Reprinted, by permission, from Ref 9.

New Compositions

While the above advances continue it has been recognized that totally new chemistries are required to broaden the range of composite possibilities. As shown in Figure 12, Professor Okamura (Ref 8) has used the current polycarbosilane-based chemistry to produce Si-N-O and Si-N fibers through a change in high temperature heat treatment atmosphere. In addition he has investigated the use of electron irradiation curing on these fibers, which caused some increase in fiber tensile strength. While having been developed during the past 2 years, these fibers have apparently not yet been chosen by a company for commercial production.

A different polymer chemistry has been used by Dr. Takeshi Isoda and his coworkers at Tonen Corporate R&D (Ref 10). They have succeeded on a pilot scale level (Table 7), and it can be expected that their fiber will become available in limited quantities some time in 1989. It has been demonstrated that this fiber has excellent potential for stability up to at least 1,200 °C and they continue to explore routes to performance improvements.

While few other specifics were learned it appears that several other organizations in Japan are in the process of developing fibers with Si-C and B-N type chemistries. Another route to different reinforcement chemistries is whisker growth. While SiC, Si_xN_4 and C whiskers are currently available in Japan, it appears that many other compositions are under development. Whisker development in the United States has placed emphasis on high performance for MMCs and high temperature stability for CMCs. In Japan the interest in also achieving very low cost can be expected to stimulate a wider variety of fabrication approaches and chemistries during the next few years. As noted previously, the diameters of whiskers will be increased to over 2 microns to lessen any potential health hazards.

METAL MATRIX COMPOSITES

BACKGROUND

Japan's involvement in research on metal matrix composites seriously started about a decade ago, a relatively short time span compared to an involvement in the United States and Europe that dates at least twice that far into the past. What seems to have motivated Japan to enter the field at that time is a combination of (1) the attainment by Japan of a position of prominence in research, development, and production of fibrous and whisker reinforcements compatible with medium melting point matrices, aluminum and magnesium; (2) demonstrated feasibility of the metal matrix composite concept by research mostly performed in the United States and Europe; and (3) the advent of an economical process for producing MMC parts, namely casting, most usually in the form of squeeze casting. Japan's enthusiasm for these materials was further enhanced by the commercialization of Art Metal's squeeze-cast, selectively reinforced aluminum diesel engine piston used by Toyota.

The structure of the R&D community in metal matrix composites in Japan is such that most of the funding and the research emanates from industry. Japan's industrial community is reported to fund about three to four times as much R&D in metal matrix composite materials than the Government (Ref 11). Many companies have entered the arena with varying motivations. Government involvement is in the form of (1) the MITI R&D Project for Basic Technology for Future Industries, which sponsors research on fiber-reinforced metals in industrial laboratories, national laboratories, and universities (see REVIEW OF JAPAN'S ADVANCED COMPOSITE MATERIAL PROGRAM) and (2) "normal" Government funding of universities and national laboratories. This is in strong opposition to the United States and Europe, where most research was (and is) funded by the Government. A further difference is the absence in Japan of a very large aerospace industrial base, which has influenced considerably the nature of research on metal matrix composites, rendering cost a more pronounced concern than in the United States or Europe. Hence, in particular, there is the very strong emphasis on casting processes.

Japan has risen very rapidly to a position of worldwide prominence in fiber- and whisker-reinforced metals technology. The first mass-produced metal matrix composite application is a Japanese achievement, as are several of the most exciting engineering innovations, to be reported below. Japan has performed pioneering research in the solidification processing of reinforced metals enabling it to lead in MMC squeeze casting technology. Another strong point of Japanese research and development of metal matrix composites is that it is strongly linked with research on fiber and whisker technology. This is due to Japan's prominence in fiber and whisker production and development and allows rapid progress in customizing reinforcement production for metal reinforcement.

Comparatively, the United States and Europe lead in applications of metal matrix composites in areas where cost is presently of little concern: aerospace and defense. Also, interest in particulate-reinforced metals is nearly nil in Japan, most likely because there is at present virtually no primary aluminum production in that country due to the energy-intensive nature of that industry and because of the large number of whisker producers in Japan. Comparatively, particulate-reinforced metals have recently been the focus of much industrial interest in metal matrix composites in the United States and Europe, where definite technological prominence has been achieved over the past years on these materials.

INSTITUTIONAL CAPABILITY— RESEARCH, MATERIALS, PRODUCTION

Industrial companies engaged in R&D in metal matrix composites can be broken down into (1) reinforcement producers trying to provide outlets for their fibers or whiskers; (2) potential users trying to gain expertise in the application of metal matrix composites, a group comprising automobile manufacturers and Japan's aerospace companies; and (3) materials companies seeking to diversify. These are listed in Table 8. All of these companies have produced metal matrix composites on a laboratory scale, and a few have actually tested parts in applications, while two (e.g., Honda and Toyota) have commercially produced metal matrix composites. The number of people involved in research on composite materials in each of these companies varies from about 2 to a total of over 15 research engineers within the Toyota Group.

Several of the prominent university and national laboratories in the field are listed in Table 9. These, as well as companies shown in Table 8, are listed based on the author's knowledge of the field. These tables are, therefore, neither exhaustive nor a listing of research groups we visited during this Office of Naval Research study (these are indicated by an asterisk in the far right column of the table). The structure of that section of the MITI R&D Project for Basic Technology for Future Industries relevant to metal matrix composites is given in Figure 2. This project expires at the beginning of 1989 and is to be replaced by a new 8-year project in April 1989 that will be predominantly on elevated temperature composites for aerospace applications. Leaders in the field are very hard to determine because the structure of research in metal matrix composites in Japan is based on a large measure of competitiveness, both within industry and within the MITI project. This, together with the specificity of research on metal matrix composites in Japan (e.g., concern with cost and stronger emphasis on nonaerospace applications), has led to significant duplication of research. For example, a large fraction of the industrial research groups listed Table 8 is focusing its attention on manufacturing metal matrix composites by squeeze casting, and many manufacturers have investigated the same applications: connecting rods, piston pins, pistons. Most of this work is of very high quality from a technological standpoint. There is, thus, a collective strength in specific areas, together with some brilliant engineering innovations emanating from a few groups that therefore stand out as such. These will be given in the next section.

Table 8. List of Companies Having Undertaken R&D Work on Metal Matrix Composite Materials, with Indication of the Nature of the Work[a]

Company	Main Areas of Research	Reference
Art Metal Manufacturing Co.	Manufacture of automotive components	12
Fuji Heavy Industries	Solid state processing, including roll diffusion bonding	13
Honda R&D Co.	* Stainless steel reinforced Al connecting rod (possibly discontinued) * Carbon fiber/Mg	14
Hitachi Research Lab	Carbon fiber-reinforced Cu by electroplating and hot pressing	15
IHI	Silicon carbide fiber-reinforced titanium by foil diffusion bonding	
Kobe Steel[b]	SiC whisker-reinforced aluminum by P/M or pressure casting, followed by mechanical working, silicon carbide fiber-reinforced titanium by powder metallurgy	16
Mitsubishi Aluminum	Pressure-infiltrated and extruded SiC whisker/Al alloys	17
Mitsubishi Heavy Industries[c]	* Pressure-infiltrated and extruded SiC whisker/Al alloys * Consolidation of C/Al and Nicalon/Al preform wires manufactured by Toray and Nippon Carbon, respectively * Ti matrix composites by HIPing	18
Mitsubishi Electric[c]	Squeeze-cast SiC whisker-reinforced Al for space applications	
Nippon Carbon[c]	Al/Nicalon preform wire	19,20
Nippon Denso	* Whisker-reinforced Cu * Tribological material: SiC/WS_2/Al	21

(continued)

Table 8. Continued

Company	Main Areas of Research	Reference
Nippon Kokan K.K.	Squeeze-cast alumina/Al	
Nippon Light Metal	* Al infiltrated 3-D networks of short alumina fibers for subsequent casting * SiC coatings on carbon fibers for MMC applications	22
Nippon Steel[b,c]	* Carbon, SiC, alumina fiber-reinforced aluminum alloys by squeeze casting * Joint project with Fuji Heavy Industries on MMC skin ¬aterials for hypersonic airplanes * Particulate-reinforced aluminum and steel by compocasting * Copper-infiltrated carbon for electrical applications	
Showa Aluminum	Mechanically alloyed SiC/Al and alumina/Al with particle sizes $<1\ \mu m$	11
Showa Denko	* RSP Al-Si reinforced with 5-10% fine ceramic * "SXA" SiC whisker-reinforced Al	11 23
Sumitomo Chemical Co.[b,c]	Alumina-15%silica fiber-reinforced aluminum alloys by squeeze casting (discontinued)	24
Toho Beslon	Ion-plated C/Al tape (discontinued ?)	
Tokai Carbon Co.	P/M and pressure-cast SiC whisker/Al	25
Toray[b]	* C fiber-reinforced Al by squeeze casting * C/Al preform wires by the TiB process * C fiber-reinforced Cu and Sn	26
Toshiba[c]	Squeeze-cast SiC whisker/Al	d

(continued)

Table 8. Continued

Company	Main Areas of Research	Reference
Toyoda Automatic Loom Works[c]	Squeeze-cast composites with oriented short fiber or whisker preforms using an electric field	d
Toyota Central R&D Laboratory[c]	* Squeeze-cast C/Al, SiC/Al, and alumina/Al * R&D on hybrid composites	27,28
Toyota Motor Corporation[c]	* Squeeze-cast short fiber-reinforced Al * Applications oriented, nondisclosed	29
Ube Industries[b,c]	* Squeeze-cast Tyranno/Al and silicon nitride whisker/Al * R&D on hybrid composites in collaboration with Toyota CRDL * Plasma-sprayed Al/Tyranno tape	30

[a]Based on the ONR mission to Japan, September 1988. Cited references and personal contacts of the authors. References are among the most recent emanating from or in connection with the institutions listed and are given for indicative purposes only. Warning: This list is not exhaustive!
[b]Reinforcement producer.
[c]Visited.

Table 9. List of University and Government Laboratories Active in Research on Metal Matrix Composite Materials, with Indication of the Nature of the Work[a]

Institution	Nature of Research	Reference
Hiroshima University	Theoretical & experimental work on squeeze casting, on statistical strength of MMCs	31
Hokkaido University	Fabrication and properties of chopped carbon fiber/aluminum composites	
Kyoto University	Preparation and strength of FRMs, in particular as influenced by interfacial coatings	32
Osaka University	Interface chemistry, joining	11

(continued)

Table 9. Continued

Institution	Nature of Research	Reference
Tokyo Metropolitan University	Solid state fabrication of SiC/Al by superplastic deformation of the matrix	
Tokyo Institute of Technology[b]	Mechanical, thermal, and elastic properties of fiber-reinforced metals, processing of MMCs, in-situ MMCs	33
Tokyo University[b]	Mechanical properties of MMCs, in particular fatigue and impact properties, processing of MMCs by spraying and hot pressing, interfacial chemistry, aluminum powder slurry composite fabrication	
Toyohashi University of Technology	Pressure-cast SiC whisker/aluminum	34
Toyota Technological Institute[b]	Metal matrix composites by low pressure plasma spraying, by co-spraying, by infiltration; work on whisker-reinforced aluminum, on steel wire reinforced magnesium	
Waseda University	Pressure casting of MMCs (discontinued ?)	
GIRI Nagoya	Pressure casting of whisker- and fiber-reinforced aluminum, P/M processing of whisker-reinforced aluminum	13,35
GIRI Osaka	Coatings on carbon fibers for reinforcing aluminum	36
GIRI and University of Kyushu	Pressure casting of MMCs (discontinued ?)	
National Research Institute for Metals, Naka-Meguro, Tokyo	Coatings on fibers for MMCs, tribology	

[a]Based on the ONR mission to Japan, September 1988. References in the scientific press marketing literature and personal contacts of the authors. References are among the most recent emanating from or in connection with the institutions listed and are given for indicative purposes only.
[b]Visited.

Theoretical work, in comparison, is much less extensive than in the United States or in Europe. There have been first-rate studies of the elastic, thermal, and mechanical properties of metal matrix composites (e.g., Tokyo Institute of Technology, Tokyo University); the processing of cast composites (e.g., Hiroshima University, Waseda University, Government Industrial Research Institute (GIRI) Nagoya, GIRI Kyushu, University of Kyushu); the influence of interfacial layers on mechanical strength (e.g., Kyoto University); the role played by the matrix in composite properties (e.g., Sumitomo Chemical, Toyota Central R&D Laboratory, Nippon Steel R&D Laboratory #1); and several other topics, but the emphasis is very clearly on engineering, and most of the research is therefore often Edisonian. The payoff is the leadership position Japan has achieved in a very short time in technical innovations in metal matrix composite processing.

MOST SIGNIFICANT ADVANCES– RESEARCH EMPHASIS

As mentioned above, there is significant duplication of research in metal matrix composites in Japan, with much of the research focusing on processing by squeeze casting. There seems to be a consensus on the nature of the procedure to be adopted, namely the use of large pressures, above 50 MPa and generally around 100 MPa. Know-how differs from one laboratory to another in the choice of optimal fiber temperature. Metal superheat is usually below 100 °C, and die temperatures are most often between 250 and 350 °C. Fibers are generally packed into steel containers, or if shaped into a handleable preform, simply dropped into the die. Essential to the process is the use of pressure to feed shrinkage and induce rapid solidification to prevent chemical reactions between fiber and matrix. For example, Japanese researchers, in particular at Toyota R&D Laboratory, have been able to infiltrate uncoated carbon fiber bundles without significant fiber degradation, both with HM fibers and HS graphite fibers. Reasons for this rather uniform choice of processing method stem from several causes: (1) the existence in the early 1980s of high quality academic research on squeeze casting; (2) concern with cost and, in particular, the demonstration of its economical viability by the mass production of the Toyota piston; and (3) simplicity of the process, allowing to overcome difficulties associated with metal shrinkage, poor wetting, and interfacial reactions. Competition for this process exists in the form of alternative casting processes using lower pressures (e.g., the Cray process) which, because of the less heavy tooling required, can be used to make larger and more intricate components. These processes were dismissed by several Japanese researchers as leading to poor infiltration and poor bonding of the fiber to the matrix. The question of which processing route is optimal still remains open, however, given the fact that our understanding of the infiltration process is still incomplete. Compared to the United States, slightly less emphasis is placed in Japan on wetting agents as a means of reducing pressures required. Alternative processes to casting exist in the form of solid state processing such as preform wire or tape consolidation, but they suffer from the point of view of cost competitiveness. These have been explored, mostly within the framework of the MITI program, but much less than direct casting processes, such as squeeze casting.

A second area where significant duplication of research exists is in optimization of matrix composition and microstructure. Several groups have empirically investigated matrices of pure aluminum, or aluminum alloyed with silicon, copper, magnesium, nickel as well as a number of other additives. They have noted vast differences in tensile strength, both at room temperature and at elevated temperatures, with both predominant types of composites, fibrous and whisker reinforced. Predominant in this area of research are the groups at Sumitomo Chemical, Toyota Central R&D Laboratory, and Hiroshima University as well as at Nippon Steel, Ube, and Nippon Carbon. The group at Sumitomo also investigated various heat treatments for these alloys, which led to spectacular differences, both in microstructure and resulting composite properties. The effect of matrix composition is related on a qualitative basis to interface reactions and interface strength, as well as to the size and distribution of second phase precipitates in the matrix. At Ube, alloying additions to the matrix added 100 °C to the useful temperature range parallel to the fibers of a Tyranno SiC hybrid/ aluminum alloy matrix squeeze-cast composite.

There is also a certain amount of work on fabrication of metal matrix composites using preform wires of fiber-reinforced metal. These materials were investigated largely as part of the MITI program, the property targets of which seem to have been met. The companies interested in these processing routes include Nippon Carbon, Ube, Toray, Fuji Heavy Industries, Mitsubishi Heavy Industries as well as researchers at Tokyo University and GIRI Osaka. The nature of the work--as well as much of its funding--is much closer to similar research in the United States, and in fact some of the work was a duplication of

the titanium diboride coating work done in the United States for carbon fiber-reinforced aluminum. Worthy of notice in this area is the fact that properties of Nippon Carbon's Nicalon-reinforced aluminum wire have been vastly improved, thanks in part to the use of an Al-5%Ni alloy as a matrix, which has in particular improved its strength from 1.1 to 1.5 GPa. An elegant preform wire consolidation method was also exposed to us at Mitsubishi Heavy Industries. The method consisted of rapid heating of the composite wire/metal foil stacking, followed by rapid transfer to a press and hot pressing in the partly liquid state with relatively cold platens, which allowed for simultaneous consolidation and solidification of the composite, thus minimizing matrix/fiber chemical interaction ("warm platen method").

Metal matrix composite parts have been tested in service by several organizations, and examples include specialty bolts, fan blades, jet engine impellers, helicopter transmission housing, spinning machine parts, power semiconductor devices and, of course, various connecting rods, pistons for diesel engines, and piston pins (Ref 31). Mitsubishi Electric intends to use silicon carbide whisker-reinforced aluminum joints for space truss applications in the near future. Emphasis is placed on commercial applications, versus aerospace and defense in the United States. Only one of these, the Toyota piston, had reached full production to nearly 20,000 pistons per month.

Among the innovations that really stood out as completely new and important, two were prominent. One is the production by Ube of a "hybrid" SiC Tyranno fiber, following initial research at Toyota Central R&D Laboratory by S.I. Yamada and S.I. Towata. The basic idea is that if each fiber is covered by a small amount of very fine ($<1\,\mu$m) silicon powder, the fibers will not touch in the composite. This, in turn, will

increase significantly the transverse strength because any transverse crack will have to pass through the matrix rather than travel from one touching fiber to another through the composite. Voids at fiber contact points are also suppressed, some degree of control of fiber volume fraction can be exerted, and the longitudinal strength is also increased at the same volume fraction by suppression of crack propagation from one fiber to another via contact points, which allows some degree of fiber pull-out. A further advantage that was found to the process is that the presence of the fine silicon carbide along the fibers leads to the formation of increased amounts of aluminum oxide by reaction of the metal with the silica on the surface of the silicon carbide. This alumina in turn allows for some protection of the fibers against chemical degradation by the metal during the casting process. It is also claimed that the presence of the fine silicon carbide powder decreases the corrosion rate of the composite. This process, only a few years old, has already been put into industrial practice, has been awarded this year the ID 100 prize, and stands out as an improvement in composite processing at little added cost to the fibers, at least at their present price level. Examples of property improvements achieved by this process are given in Figure 16.

A second innovation worthy of special mention was a new process, invented at Toyoda Automatic Loom Company, for orienting short reinforcements with negligible fiber or whisker breakage. This process relies on the application of high voltages to short fibers or whiskers suspended in a nonconducting fluid to which a dispersant has been added. The fibers orient themselve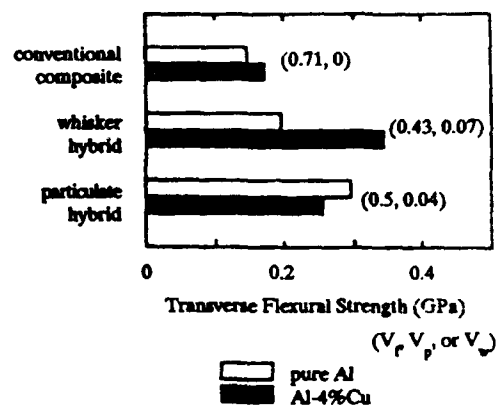s perpendicular to the equipotential surfaces, allowing the fabrication of a preform in which the fibers are preferentially oriented along one direction or in a variety of configurations, radially from a straight axis, for example. Characterization of composites produced by infiltration of such preforms showed significantly increased properties compared to random two-dimensional (2-D) or 3-D fiber orientations, as should be expected. Characterization work on these composites is presently being undertaken in the R-CAST group of the University of Tokyo as well. The prime advantage of the process is that it allows one to obtain composites with improved longitudinal strength and modulus from relatively inexpensive reinforcements like Saffil™ and it is a versatile method for manufacturing preforms of such reinforcements tailored to the component prior to casting. Some data from this work are given in Figures 17 to 19. In addition to casting, short fiber aluminum metal matrix composites are being produced via a powder metallurgy, hot extrusion approach with tensile strengths of 780 to 880 MPa (80 to 90 kgf/mm²).

FUTURE DIRECTION AND TRENDS

The Japanese approach to research and development on metal matrix composites has led to the attainment by that country of a position of prominence in squeeze-cast whisker- and fiber-reinforced metals. Reasons underlying this choice of an area of excellence were given above: industrial nature of funding, domestic whisker and fiber production, absence of an aerospace outlet, and drive for commercial applications.
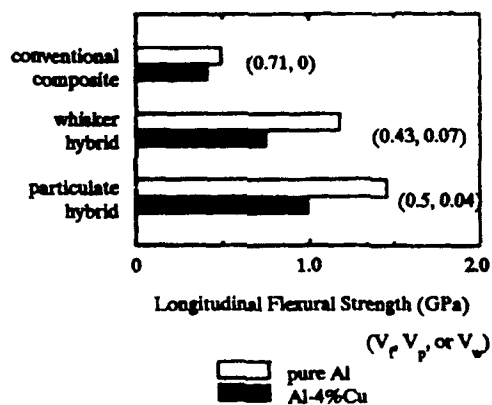
(a) Longitudinal flexural strength (GPa).   (b) Transverse flexural strength (GPa).

Figure 16. Flexural strength of parallel Tyranno Si-C-Ti-O fiber-reinforced aluminum composites produced by squeeze casting at Honda Central R&D Laboratories. Reprinted with permission from *Scripta Met.* 21, I.W. Hall, Copyright 1987.
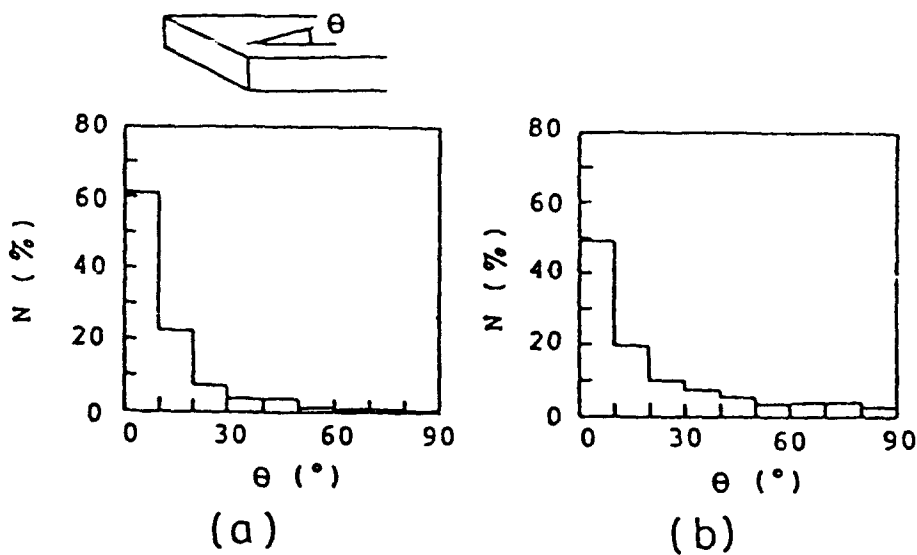

Figure 17. Orientation of discontinuous fibers or whiskers within different preforms (a,b) prepared by the electrostatic method invented at Toyoda Automatic Loom Works, Ltd. Courtesy of T. Ito.
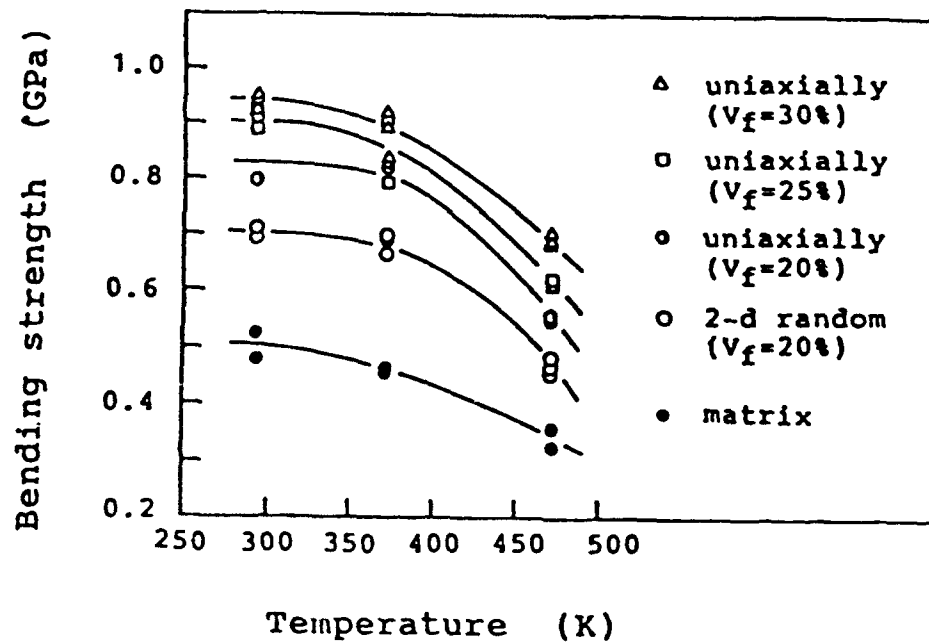
Figure 18.   Bending strength of squeeze-cast aluminum matrix (alloy JIS AC1A)
composite, reinforced with short alumina fiber preforms prepared at
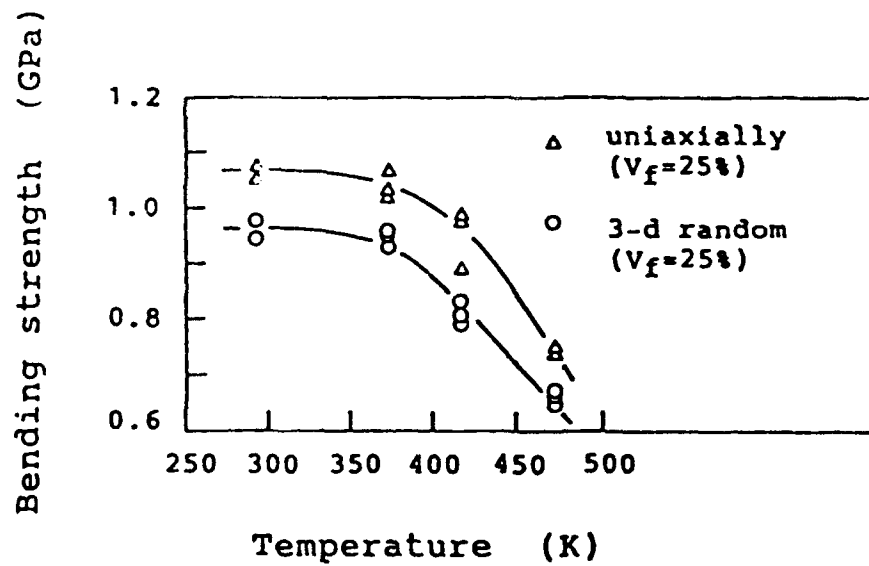Toyoda Automatic Loom Works, Ltd. Courtesy of T. Ito.

Figure 19.   Bending strength of squeeze-cast aluminum matrix (alloy JIS AC1A)
composite, reinforced with SiC whisker preforms prepared at Toyoda
Automatic Loom Works, Ltd. Courtesy of T. Ito.

Japan is planning to increase its involvement in the space and aeronautical industries in the coming years, thus rendering one of the premises of the present orientation of their research less true. There were numerous indications of this coming change during our visit, to start with the implementation of the next MITI program next April. General guidelines of that program are still being decided at this date, but it is very clear that stronger emphasis will be placed on elevated temperature composites. Several research groups are thus planning to initiate research (and in some cases have already done some preliminary work) on titanium and aluminide matrix composites.

Concomitantly, although test applications have met or exceeded targets, several barriers remain to be overcome before new applications of fiber-reinforced metals start to appear: (1) the price of whiskers and fibers is too high for most commercial applications, at least within the automotive arena where costs should not exceed about $20/lb [according to researchers at Toyota (see Figure 4)]; (2) on two occasions difficulties with convincing designers to work with composites when isotropic metals "do the job" were cited; and (3) variability in composite mechanical properties. Of these, the first is, of course, foremost, since the second has been solved for polymer-reinforced composites and the third can be solved by better understanding of processing. There were in some research groups slight signs of tiredness, perhaps due to overly optimistic expectations based on the Toyota piston example, perhaps due to the fact that more novel technological advances are being looked at: elevated temperature metal and ceramic matrices (in part due to the coming MITI program and to Japan's increasing interest in aerospace industry) and elevated temperature polymeric matrices for temperatures around 300 °C.

A slight shift of focus toward these new materials is to be expected in the future, both in industry, governmental laboratories, and universities--if only because they are new and thus generate enthusiasm among researchers. This is an important element in a country where industrial R&D aims farther into the future and is planned with more say by engineers and scientists and less by accountants than in most other countries. It is also becoming somewhat clearer than it was 2 years ago that metal matrix composites are still largely materials of the future and that their application in the commercial civilian world will be a slower process than some have anticipated based on the Toyota piston success story. One company (Sumitomo Chemical) has thus, after much very good research, decided to stop working on MMCs until it can find a metal matrix application of its alumina-silica fiber (we were told that this decision was taken in part based on a change of location of the company's research laboratory and the high cost for moving a large squeeze casting press for research purposes only). Japan's commitment to metal matrix composites remains high, however, and is expected to remain so in the coming years. Several companies and research laboratories (Ube and Toyota, for example) have research groups of over 10 engineers working on these materials, with no plans to cut back on that level of investment in MMCs.

Areas to be followed in the future will thus be (1) innovations in the processing of metal matrix composites, including consequences of technologies such as the method for orienting preforms cited earlier; (2) possible nearer term high cost/high payoff applications in aerospace, electronic, or industrial machinery, for example, including results from research on elevated temperature metal matrix composites performing within the range of goals set by the Department of Defense; (3) changes in the reinforcement market based on technical innovations and increased demand from the polymer-reinforced composites industry; and (4) Japan's reaction to an offer of inexpensive particulate-reinforced aluminum, in particular by ALCAN.

CERAMIC COMPOSITE RESEARCH IN JAPAN

In this section the current status and scientific accomplishments of university, government labs and, to a lesser extent, selected industrial labs in ceramic composite research a.e summarized. As discussed in more detail below, much of the effort is devoted to SiC whisker-reinforced ceramics. The first section reviews the various ceramic matrices for which SiC whiskers were added, the conditions under which they were fabricated, and the best mechanical properties obtained. The second section deals with borides and silicide composites. Fiber-reinforced composites are dealt with separately. The final section overviews the major new facilities devoted to ceramic research in Japan.

SiC WHISKER-REINFORCED CERAMICS

Alumina

E. Yasuda et al. (Ref 37) at the Tokyo Institute of Technology studied the effect of diameter, length, and aspect ratio of SiC whiskers on the bend strength and fracture toughness of 20 vol % SiC$_w$/alumina composites (Figure 20). The whiskers were deagglomerated ultrasonically and mixed with the alumina powder in butanol and hot pressed (1,700 to 1,800 °C, 33 MPa, 1 hour). From initial data, the toughness appears to increase linearly with both the length and diameter of the whiskers as shown in Figure 20b; however, newer data showed no relation with whisker length and fracture toughness. The bend strength, however, decreased as shown in Figure 20a. The degradation in strength was found to correspond well with the whisker length, leading to the conclusion that the critical flaw size scaled with the length of the reinforcing whisker. Furthermore, the addition of whiskers seemed to inhibit grain growth in alumina as well as enhance the properties at higher temperatures.

Niihara et al. (Ref 38) investigated the effect of 2- and 0.3-μm SiC particle additions to alumina, and in general it was found that the fracture toughness and strength improved with the dispersions. The samples were made by hot pressing (1,500 to 1,800 °C, N$_2$ atmosphere, 28 MPa) of the powder mixtures. The best results were obtained for a 5 vol % dispersion of 0.3-μm β-SiC where a fracture strength of 1,100 MPa and a toughness of 4.7 MPa$\sqrt{m}$ were achieved. This remarkable improvement is believed to be due to the dispersion of the SiC particles within the alumina grains and the concomitant compressive residual microstresses that result in the matrix.
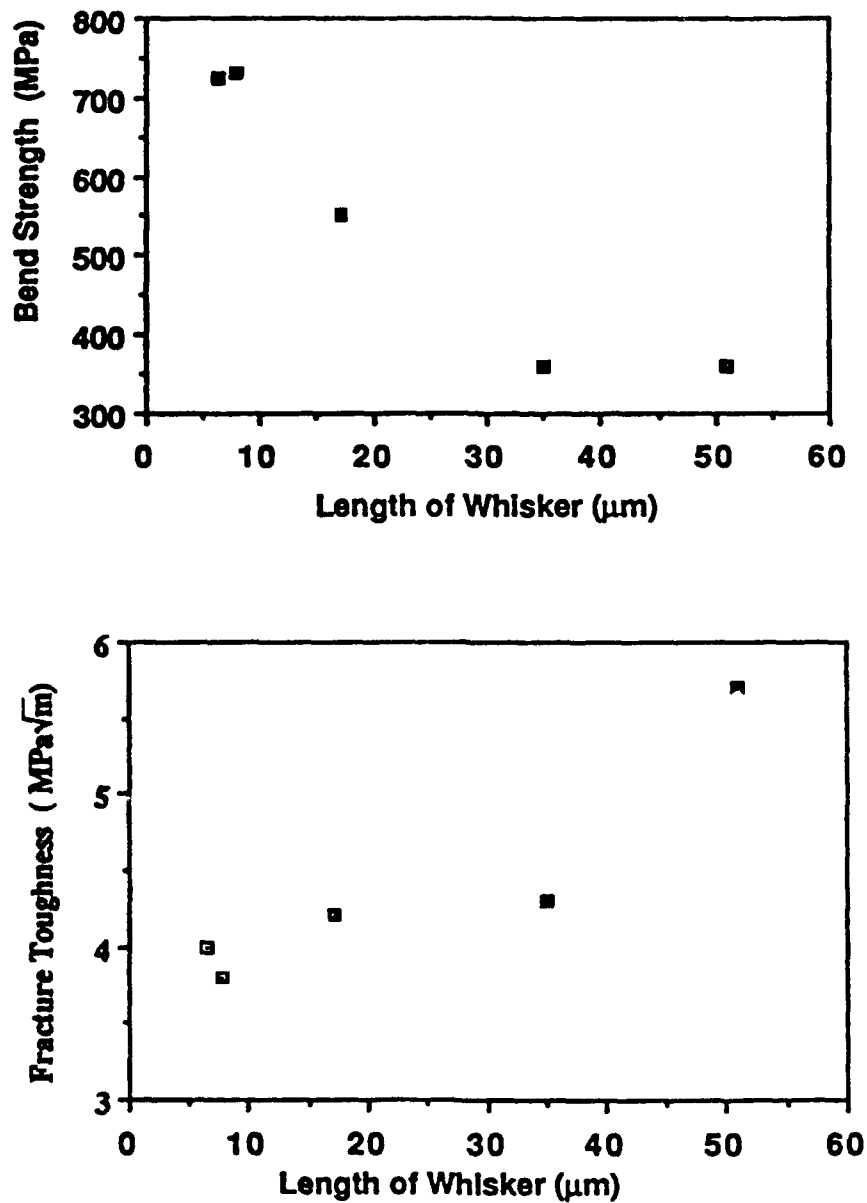
Figure 20. Dependence of flexural strength (a) and fracture toughness (b) on length of whisker. "Development of SiC whisker reinforced alumina," by E. Yasuda et al., *Proc. of International Congress on Advanced Composites for New Systems and Technologies* (1988). Adapted by permission of the publisher, Simmy Schnabel.

Silicon Nitride

Since the introduction of the Si_3N_4 turbocharger rotors in Nissan's cars, a second-generation rotor that provides even greater reliability and higher performance has been developed (Ref 39). Several automotive companies in Japan are currently investigating toughening of Si_3N_4 with SiC whiskers. Nissan Motor Co. found that it was difficult to sinter the Si_3N_4 with the addition of the SiC whiskers, and while a 40-percent increase in fracture toughness was observed, the bend strengths were found to decrease. Toyota and Toshiba are also working on the same system and some of their results were reported at the Japan Ceramics Society Spring 1988 Meeting.

Starting with amorphous Si-C-N powders chemical vapor deposited from a $[Si(CH)_3]_2NH-NH_3-N_2$, followed by hot pressing (N_2 atmosphere for 3 hours at 1,700 to 1,800 °C), Niihara et al. (Ref 40) obtained room temperature flexural strengths of 1,000 MPa. The toughness was found to peak at about 15 vol % SiC with a K_{Ic} of 7 to 8 MPa$\sqrt{m}$ as shown in Figure 21. The SiC particles were found to be dispersed not only in the grain boundaries but within the Si_3N_4 grains. Below 10 vol % the SiC dispersions accelerated the growth of elongated Si_3N_4 grains, which is believed to be responsible for the improved toughness. The degradation in strength as a function of temperature is shown in Figure 22.
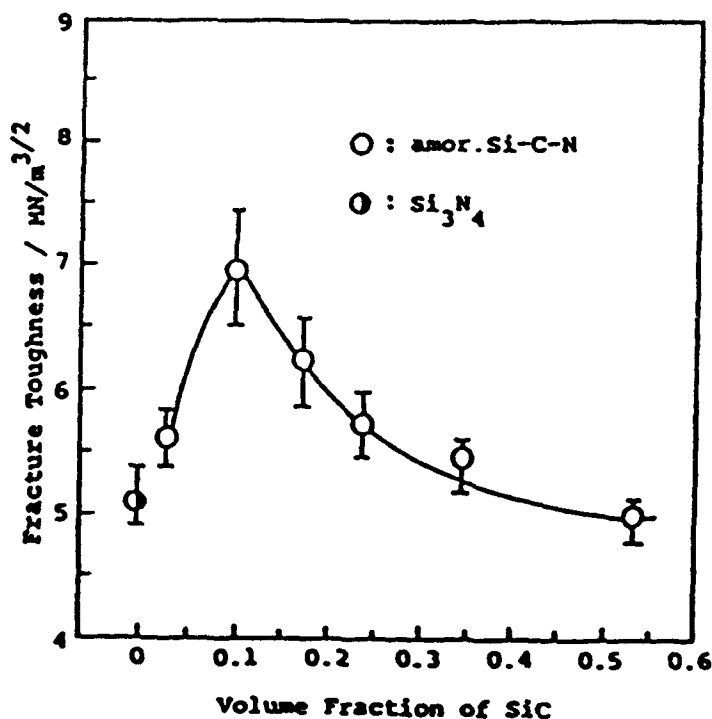

Figure 21. Effects of SiC dispersions on the fracture toughness of composites prepared from fine, amorphous Si-C-N powder. Reprinted, by permission, from Ref 40.
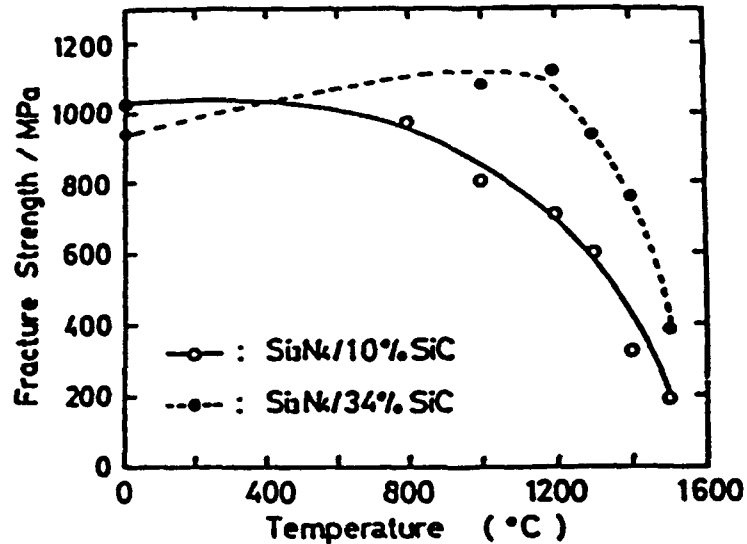
Figure 22. Variation of fracture strength with temperature
for Si_3N_4-SiC composites prepared from fine,
amorphous Si-C-N powder precursors.
Reprinted, by permission, from Ref 40.

Yamada et al. (Ref 41,42) are using another innovative approach to reinforcing Si_3N_4 with SiC whiskers, whereby the whiskers are formed in situ. Carbon black, α-Si_3N_4, silica, $CoCl_2$ (catalyzer), and NaCl (space-forming agent) are mixed and heated at 1,600 °C for 1 hour. After the excess carbon is burned off, the sintering aids added, and the deagglomeration of the mixture by milling carried out, the mixture is hot pressed.

Compared to physically mixed powders, the bulk density was improved but the bend strength was lower, most probably due to residual carbon. The advantages of this process are its economy and the reduced health hazards associated with the handling of very fine SiC whiskers. It should be noted here that several of the groups working with SiC whiskers voiced their concern about possible health hazards associated with the very fine whiskers.

At Osaka University, SiC whisker (Tokai Carbon, Tatako) reinforced Si_3N_4 (powder supplied by Ube Industries or H.G. Stark) is HIPed without additives. The Ube Si_3N_4 is formed by the decomposition of a Si amide and is of very high purity. It is dispersed ultrasonically with a dispersion agent. The SiC whiskers are $0.5\,\mu$m in diameter and $30\,\mu$m in length. After being ultrasonically agitated, the mixtures are stored in a polycarbonate dispersant so as to further promote their dispersion. Material with 20 vol % SiC whiskers exhibited a four-point flexural strength of about 500 MPa, whereas the same material with 30 vol % SiC whiskers showed a higher flexural strength at room temperature but a lower flexural strength at 1,400 °C. In general, the dispersion of the whiskers has been difficult due to their agglomeration.

The Government Industrial Research Institute at Osaka (GIRI Osaka) has fabricated and tested SiC whisker-reinforced Si_3N_4 matrices. The fabrication method involved mechanically dispersing the whiskers in water and removing the SiC lumps by sieving. The Y_2O_3 (sintering aid) doped Si_3N_4 was ultrasonically dispersed and the two dispersions were mixed, stirred, and filtered. The green plates were then hot pressed (1,800 °C, 34 to 39 MPa, N_2 and CO, 15 to 90 minutes.) Because needlelike whiskers prevent shrinkage of the green body, the sintering rate for SiC fiber-reinforced composite in a Si_3N_4 matrix was less than that for monolithic Si_3N_4. Thus it was critical to choose a sintering (or hot pressing) time that was long enough to obtain a fully dense body but was also short enough to minimize the reaction between whisker and matrix. This time has to be experimentally determined for the particular characteristics of the SiC whisker. In the present instance, where nearly all of the β-SiC whisker had a diameter between 0.2 and 0.5 μm, a length of 20 to 50 μm, and an aspect ratio of 50 to 300, and the α-Si_3N_4 had an average particle size of 1 μm and contained 1.82 percent O and 0.92 percent C, the optimum hot pressing time for achieving the best fracture strength at 1,300 °C was 60 minutes. The fracture strength at 1,300 °C was maximized at about 20 wt. % SiC whisker content and decreased at higher whisker concentrations due to an increased concentration of glassy phase at the grain boundaries.

In an earlier, separate study, Si_3N_4-SiC whisker composites with SiC whisker content up to 30 wt. % were prepared by heating at 1,700 to 2,000 °C for 60 minutes under 1 MPa of N_2. Fully dense composites were obtained at 2,000 °C. The optimum content of the sintering aids, Y_2O_3 plus La_2O_3, was 20 and 30 mol % for the whisker contents of 10 and 20 wt. %, respectively. Room temperature bending strengths were 596 and 560 MPa for 10 and 20 wt. % additions of whisker, respectively. Moreover, these composites had strengths that were more than 80 percent of the room temperature value at 1,300 °C. An improvement in the strength was realized by using a sieved whisker.

The fracture toughness for Si_3N_4 composite containing 0 to 30 wt. % SiC whisker was measured by indentation microfracture (IM) and Chevron notch (CN) methods. The results obtained by the IM method showed that K_{1c}, calculated on the basis of a Palmquist type crack, varied from 6.3 MPa$\sqrt{m}$ for the Si_3N_4 matrix to 7.2 MPa$\sqrt{m}$ for the composite containing 15 wt. % whisker. On the other hand, K_{1c} obtained from the CN method varied from 5.1 MPa$\sqrt{m}$ for the Si_3N_4 to 6.3 MPa$\sqrt{m}$ for the composite having 30 wt. % whisker. Microscopic observation of the cracks induced by indentation showed that the crack propagation is often inhibited by the whisker and the matrix. This crack deflection may be attributed to the stress fields set up at the interface due to thermal expansion differences between the whisker and matrix.

GIRI Osaka is also investigating electric discharge machining of SiC whisker-reinforced Si_3N_4 and has carried out pioneering studies on the development of simple and reliable methods for the joining of ceramics (Ref 43).

β-Sialon

The conventional way of fabricating sialon ceramics involves the reaction of mixed powders of Si_3N_4, Al_2O_3, and AlN, or Si_3N_4, Al_2O_3, and SiO_2. However, flaws such as pores and coarse-grained agglomerates are often formed inside the body during the sintering process as a consequence of the extreme difficulty of homogeneous mixing

of these powders. At GIRI Kyushu, in order to obtain a homogeneous microstructure, β-Sialon was fabricated by using Si_3N_4 as a starting powder and Al-alkoxide. Hot-pressed β-Sialon made by this process had a relative density of more than 98 percent and a more homogeneous microstructure than that for conventionally fabricated β-Sialon. As a consequence the room temperature flexural strength improved from 500 to 600 MPa to 1,000 to 1,600 MPa.

The effect of additives on the mechanical properties of β-Sialon fabricated from Si_3N_4, Al_2O_3, and AlN powders was investigated. Oxide additions generally increased room temperature strength but resulted in a decrease in high temperature strength as a consequence of their promoting the formation of a glassy grain boundary phase. However, the addition of some carbides resulted in an improvement in both the room and high temperature strengths. Thus, the effects of the addition of SiC to β-Sialon are under investigation. Room temperature flexural strength increased with SiC content to about 50 vol % SiC, where it exhibited a maximum and then decreased with higher SiC concentration. Other properties such as hardness and thermal conductivity also varied with SiC concentration.

Also at GIRI Kyushu, gas pressure sintering of Si_3N_4 with concurrent additions of 0 to 5 wt. % Al_2O_3 and 5 wt. % rare earth oxide (Y_2O_3, La_2O_3, and CeO_2) was studied at 1,800 °C and under 2 to 4 MPa of N_2 pressure. A high bulk density of 3.27 g/cm^3 was obtained for Si_3N_4 with 1 or 3 wt. % Al_2O_3 and 5 wt. % CeO_2. This material was pressure sintered by a two-step method first at 1,700 °C or 1,800 °C at 0.2 MPa for 1 hour and directly heated to 2,000 °C and 4 MPa for 1 hour. The weight loss was less than 3 wt. %. The adequate minimum amounts of Al_2O_3 for the densification of Si_3N_4-5 wt. % Y_2O_3, Si_3N_4-5 wt. % La_2O_3, or Si_3N_4-5 wt. % CeO_2 were ~1.5 to 2.0 wt. %, ~1.0 to 1.2 wt. %, and ~1 wt. %, respectively. The Si_3N_4 sintered at 2,000 °C by the two-step method showed a high fracture toughness value of 9 MPa$\sqrt{m}$ due to the high aspect ratio of the needle or fiberlike microstructure. The first stage of pressure sintering is believed to develop this needlelike microstructure. The second stage then gives elongated structures that are very dense. It was also shown that the final density is dependent upon the heating rate from the first sintering step to the second sintering step, which was attributed to the differences in the amount and viscosity of the liquid phase.

Stabilized Zirconia

In addition to its work on toughening Si_3N_4 with SiC whiskers, Nissan Motor Co. (Ref 44,45) has also investigated the addition of SiC whiskers to Y_2O_3-tetragonal zirconia polycrystal (Y-TZP). Although bend strengths of 950 MPa with K_{Ic} = 8.2 MPa$\sqrt{m}$ were obtained at room temperature, the mechanical properties were severely degraded at higher temperatures due to the tetragonal to monoclinic transformation. As a result the project has since been dropped.

At Osaka University densities above 96 percent of theoretical were achieved for ZrO_2-dispersed Si_3N_4 composites that were HIPed without additives [180 MPa, 1,850 to 1,900 °C, 1 hour, glass encapsulation (Ref 46)]. A dispersion of 20 wt. % of a ZrO_2 powder containing 2.5 mol % Y_2O_3 was found to have a room temperature fracture toughness of 7.5 MPa$\sqrt{m}$ because of the high retention of tetragonal ZrO_2.

Spinel

At GIRI Nagoya, room temperature flexural strengths of 600 MPa, at V_f = 20 percent, were achieved in noncarbon-coated SiC whisker/spinel composites (Ref 47,48). The whiskers were made by Tokai Carbon (0.5 μm, 30 to 50 μm long) and the matrix was a powder (Iwatani Chemical Co., SP-12, MgO-Al$_2$O$_3$, 0.3 μm). The whiskers and spinel powder were mixed in ethanol using nylon balls in a polyethylene container. The samples were hot pressed (1,850 °C, 33 MPa, Ar atmosphere). When

tested at room temperature and 1,300 °C, the fracture toughness increased with whisker volume fraction for both carbon- and noncarbon-coated whiskers (Figures 23 and 24). The carbon coating of the whiskers promoted whisker pullout and contributed slightly to the increase in fracture toughness at higher temperatures (K_{Ic} = 10.6 MPa$\sqrt{m}$ at 1,300 °C, V_f = 0.4). The SiC whiskers were also found to suppress grain growth of the spinel matrix. At room temperature the flexural strength appears to peak at about 20 vol %, whereas at 1,300 °C the flexural strength increases with volume fraction (Figures 23 and 24).
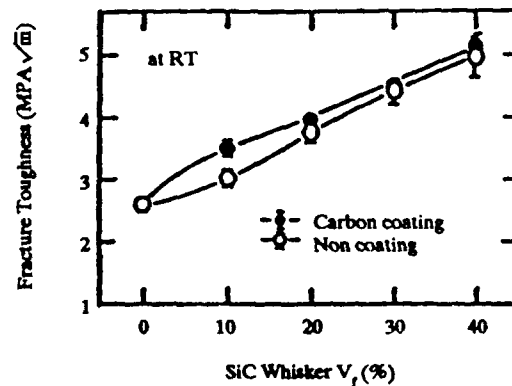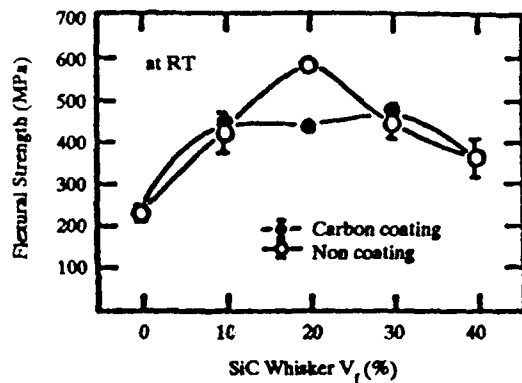

Figure 23. Flexural strength (a) and fracture toughness (b) of SiC whisker/spinel composites at room temperature. Reprinted, by permission, from Ref 48.
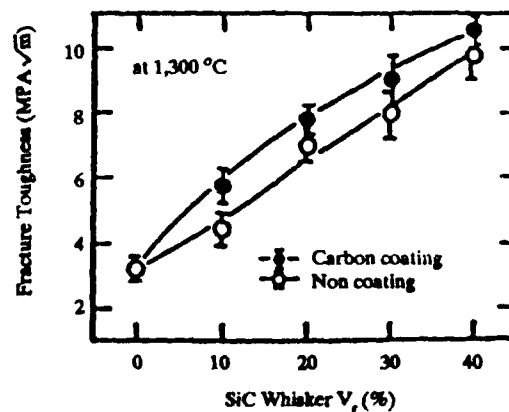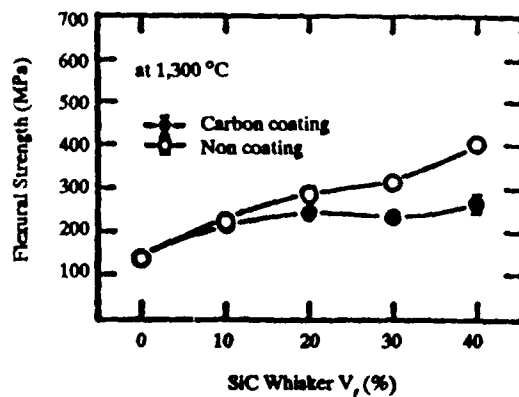

Figure 24. Flexural strength (a) and fracture toughness (b) of SiC whisker/spinel composites at 1,300 °C. Reprinted, by permission, from Ref 48.

TiB$_2$-BASED CERAMIC COMPOSITES

At GIRI Kyushu an active research program dealing with sintered mixtures of titanium borides, nitrides, and MoSi$_2$ is underway. One objective of this effort is to exploit the high temperature plastic behavior of the diboride-based ceramics by means of metallurgical forming methods. A second objective is to exploit the superior wear resistance of TiB$_2$-TiN based ceramics.

TiB$_2$-TaB$_2$-CoB-TiC Composites

The addition of 1.7 percent TiC to the base composition TiB$_2$-5%TaB$_2$-1%CoB fine powder mixture (hot pressing at 1,500 °C, 1 hour, 20 MPa in vacuum) reduced the porosity from 0.3-0.7 to 0.1-0.2 vol %. Prior to hot pressing, the average particle size was reduced, by extended ball milling, from about 4 to 1.0 μm, which resulted in an increase in the oxygen content of the powders. The lower porosity is believed to be due to a (Ti,Ta)(C,O) solid solution forming during sintering. The room temperature transverse rupture strengths of the TiC containing composites were similar to the base composition, about 1,000 MPa. Three factors that affected the transverse rupture strength were (1) lower oxygen content, which increased it; (2) larger average grain size of TiB$_2$, which decreased it; and (3) the formation of the solid solution phase of (Ti,Ta)(C,O) as indicated above, for which the relationship is not apparent (Ref 48,49).

Sintering and Oxidation of Ti(C,N)-TiB$_2$-MoSi$_2$ Composites

Ternary composites of Ti(C,N)-TiB$_2$-MoSi$_2$ were studied in order to improve the oxidation resistance of Ti(C,N)-TiB$_2$ composites (Ref 50). It was concluded that:

- Fully densified and strong compacts were obtained for the compositions Ti(C,N)-TiB$_2$-MoSi$_2$ with less than 80 wt. % TiB$_2$ and less than 60 wt. % MoSi$_2$ by sintering 1-μm green powders above 1,750 °C.

- Good oxidation resistance was observed above 1,000 °C when the concentration of Ti(C,N) was small such that all Ti(C,N) grains were surrounded by TiB$_2$ and MoSi$_2$ grains and with the MoSi$_2$ content in excess of 20 wt. %. This good oxidation resistance was attributed to the formation of a compact surface film of rutile and silicate glass by oxidation, which resulted in an oxidation-resistant grain boundary film. This grain boundary film was initially porous but became dense after a sufficient amount of silicate glass had flowed from the inner reaction sublayers to the outer surface.

- A TiB$_2$-20 wt. % MoSi$_2$ composite sintered at 1,800 °C in vacuum was found to be oxidation resistant, with a room temperature bending strength of 600 MPa, Vickers hardness of 2100, and fracture toughness (by indentation, 196 N), K$_{Ic}$, of 3.7 MPa$\sqrt{m}$.

Further work showed that it was possible to sinter a TiB$_2$-20 wt. % MoSi$_2$ composition at 1,700 °C for 90 minutes in vacuum. The resultant microstructure consisted of TiB$_2$ particles dispersed in a MoSi$_2$ matrix. It exhibited a room temperature flexural strength of 700 MPa and a 1,200 °C flexural strength of 600 MPa. Other room temperature properties were micro-Vickers hardness of 2450, K$_{Ic}$ = 3.5 MPa$\sqrt{m}$, electrical resistivity of 18.2 $\mu\Omega$, thermal expansion coefficient of 7 to 8 x 10^{-6} °C^{-1}, and density of 4.9 g/cm^3. Weight gain by oxidation was 5 to 10 mg/cm^2 at 1,000 °C in air for an unstipulated duration. This system exhibits high

temperature superplasticity. (This program was initiated on 1 April 1988.)

Ti(C,N)-TiB₂ Cutting Tool for Steel

Composites of 70 wt. % Ti(C,N)-30 wt. % TiB₂ were fabricated and shown to have a three-point flexural strength of over 800 MPa at room temperature, K_{Ic} > 5 MPa√m, H > 2500, α = 8 x 10^{-6} °C^{-1}, and exhibited excellent cutting tool behavior. The Ti(C,N)-Cr₃C₂ system is also now under investigation. Both Ti(C,N)-TiB₂ and Ti(C,N)-Cr₃C₂ system cutting tools have been verified to have a longer lifetime for the high speed (300 m/min) cutting of plain carbon steel than the lifetime for WC-Co and TiN cermet tools under comparable circumstances. These new composite materials are both now under evaluation for cutting heat-resistant and stainless steels. The Ti(C,N)-TiB₂ system does not contain any "critical" or "strategic" elements.

FIBER-REINFORCED CERAMICS AND GLASSES

Fiber-Reinforced Glass and Glass Ceramics

Two companies making composites by this approach are Mitsubishi Electric and Nippon Carbon Co. Mitsubishi Electric's interest appears to be primarily for space-based satellite applications where dimensional stability is important. The systems of interest include carbon, Nicalon, and SiC monofilament fiber (Avco) reinforced borosilicate glass, as well as SiC monofilament LAS and vycor glass systems.

Nippon Carbon Co. is mainly interested in the use of Nicalon fibers as reinforcement. They believe that the glass matrix approach can provide relatively low cost/high performance materials. The two systems they are currently making are Nicalon-reinforced borosilicate glass and LAS ceramics.

Fiber-Reinforced SiC Composites

Using a slurry infiltration method (Ref 51) followed in some cases by liquid Si infiltration, a group at GIRI Nagoya (Ref 52) is fabricating continuous fiber-reinforced SiC composites. The fibers used were carbon fibers, SiC-coated C fibers, or Nicalon fibers. The best properties [$\sigma_{3\text{-point}}$ = 277 MPa, K_{Ic} = 9 MPa√m] were obtained for a C fiber SiC matrix, hot pressed at 1,600 °C. In general, however, the level of porosity was quite high.

Polymer-Derived Ceramic Matrices

The polymer approach to creating ceramic matrices has become very popular with those companies that have the capabilities to produce polymer-derived fibers. Using essentially the same polymers they are able to infiltrate the fiber preform to convert the porous ceramic matrix. Repeated infiltrations can raise the density of the matrix. Nippon Carbon is the only company that has publicly displayed this type of composite (Nicaloceram®). The other companies are still doing their development work; however, it can be expected that they will come forward in the near future with impressive data.

The advantages of this approach are the possibility of making very large articles without the need for a hot press and the possibility of making very high temperature materials.

MAJOR NEW FACILITIES

In April 1987, the Japanese Fine Ceramics Center (JFCC) at Nagoya (cost ¥18.0B, 10,878 m²), dedicated exclusively to ceramics research, was opened. Currently there are about 60 researchers and technicians working at the JFCC. The ultimate goal is to have about 150, a third of which will come from industry for 1 to 2 years, with a maximum stay of 3 years.

The mission of the JFCC is to carry out basic and applied research on fine ceramics, establish standardized testing methods, promote mutual cooperation between universities and industry, establish a data base, and promote international cooperation. The facilities at the JFCC are quite impressive with just about every facility needed to carry out both fundamental and applied research on ceramics available.

Another major new facility that is currently being built on 160 acres 30 miles west of Tokyo is the Uenohara Research and Techno-Park Project. West Tokyo University is being built on the site, with a targeted enrollment of 1,600 students in electronics and information science, bioscience, materials science and technology, and management engineering.

SUMMARY AND CONCLUSIONS

While currently most of the whisker reinforcement is being carried out with very fine SiC whiskers, the next generation of whisker-reinforced composites will most likely use thicker and longer whiskers for two reasons: reduced health hazard and better fracture toughness. However, the problem of decreasing bend strengths as the length of the whisker increases will have to be solved.

It is most likely that the next generation of rotors or engine components will be whisker-reinforced Si_3N_4. The automotive companies will try to build on their expertise and track record with Si_3N_4. Their preferred fabrication method will probably be to use a pressureless or gas pressure sintering approach rather than hot pressing.

The results obtained with nanocomposites are quite encouraging; however, the issue of high temperature stability of the very fine microstructures has to be addressed.

In the area of fiber-reinforced ceramics two distinct approaches seem to be evolving: one based on high volume, low cost, low temperature matrices such as glass and glass ceramics and the second based on polymer-derived matrices with repeated infiltration to fabricate very high temperature performance composites, e.g., SiC/SiC or C/SiC. As mentioned above, the advantages of the latter approach are the possibility of making very large articles without the need for hot pressing and the possibility of making very high temperature materials.

FUTURE DIRECTIONS AND APPLICATION

BACKGROUND

The future development of composites technology is dependent upon cost-effective applications and design confidence. The development of high performance advanced composites in Japan is a historic development highlighted by several parallel government, industry, and academic research initiatives. The major program, an 8-year effort involving industry, government, and university research, i.e., the National Research and Development Project for Advanced Composite Materials (ACM), is concluding (Figure 2). In 1981, the ACM program was initiated under the direction of the Agency of Industrial Science and Technology. The program's goals were targets with regard to the specific property levels for various polymers and metal matrix systems as well as oxide-dispersed aluminum and titanium alloy systems. In addition, the commercial application and prototype parts were directed to the aerospace, gas turbine, and automotive sectors. An envisioned new program projects an 8-year, $320M (¥40B @ ¥125/$1) program in order to develop a design for a hypersonic commercial airplane and engine materials, with major companies such as Mitsubishi Heavy Industries, Kawasaki Heavy Industries, and Ishikawajima-Harima Industries (IHI) being involved in engine design efforts. The thrusts in structural composites as well as engine design will certainly gain attention from the commercial transport and executive aircraft sectors throughout the world.

Over the last 15 years in Japan, the development of a competitive aircraft industry has languished and the new program may serve to catalyze the industry. Mitsubishi Heavy Industries was active in the commercial aircraft market with a corporate jet, i.e., the Diamond, and commercial aircraft markets. More recently, disappointment with a government decision to base its FSX aircraft on a General Dynamics design vis a vis an entirely new Japanese design has been a compromise and has not assisted aircraft and composites processing developments in Japan. In terms of composites and advanced materials in aircraft propulsion systems, All Nippon Airlines (ANA) exercised an option for a U.S. turbine, e.g., General Electric, versus a multinational V2500 model that contained significant Japanese participation and financial support. This action will serve as a moderating influence on the growth of the gas turbine industry. In the helicopter sector, there is a joint licensing agreement between United Technologies-Sikorsky Aircraft and Mitsubishi Heavy Industries for the domestic production and distribution of the Blackhawk helicopter. Nissan, IHI, and Kawasaki Heavy Industries also have strong interest in the helicopter market. As a result, several questions concerning the future development and manufacturing of aerospace-driven composite materials occur at several application levels, i.e., private, commercial, and defense aircraft, helicopter, propulsion systems, satellite, launch system as well as the hypersonic commercial aircraft. These program requests for advanced composites and materials are in competition with program requests at MITI that include developing areas of superconducting materials, biotechnology, artificial intelligence, and deep water construction.

AREAS OF ACHIEVEMENT

At this point in time, Japan's research may be at a watershed, where significant or dominant status has been established in (1) fibers (e.g., carbon, pitch-based, and silicon carbide fibers), silicon carbide whiskers, and commercialization and utilization of pitch-based fibers in concrete structures and (2) cast metal matrix composites, e.g., production level of aluminum metal matrix composites namely the Toyota piston and the development of the squeeze casting process for fiber- or whisker-reinforced metal matrix composites. In addition, advances have occurred with the coupled development of carbon fiber precursor tow preform via metal organic chemical vapor deposition and plasma vapor deposition. Capabilities exist in several fabrication and process techniques to include hot pressing, hot rolling, laser heating and rolling, hot isostatic pressing (HIP), and squeeze casting and hot extrusion for SiC whisker aluminum composites. In the ceramic matrix composites area, Japan's efforts in the fine ceramics area with the development of excellent ceramic powders and monolithic structural ceramics combined with the fiber/whiskers development efforts are excellent initiation points for the potential development of high performance structural ceramic matrix composites.

FUTURE TRENDS

T. Hayashi (Ref 1-3) has suggested a transition from a current model of composites application with "few models and mass production" to an era of "diversified models and minor production." It appears that the transition suggests that the composites industry, particularly the polymeric and metal matrix systems, must acclimate from a few large applications to become flexible and seek diversified "niche" production techniques with a transition from labor intensive, efficient, large structures to smaller, original knowledge intensive structures with higher risks and returns. For large-scale structures, the development of composite materials may preclude a single company or country effort. As a result, the need for international cooperation, particularly with high risk programs (e.g., space structures), suggests global cooperation. To this end, a data base with standardized testing procedures yielding statistically reliable properties with design allowables is required for commercial applications coupled with reduced reinforcement costs. For future developments in the commercial sector, international cooperation will be required for data base and system development and integration.

APPLICATIONS OF POLYMER MATRIX COMPOSITES

Polymeric composite materials consumption has been estimated by the Japan Reinforced Plastics Society to be 279,000 tons/yr for GFRP with a total consumption of about 350,000 tons/yr, which can be compared to the consumption in Europe and the United States (820,000 and 1,000,000 tons/yr, respectively). Hayashi (Ref 1-3) has cited several typical structural applications, which include the short take-off and landing "ASUKA" aircraft with graphite/polyimide and Nomex sandwich structures in the upper wing skin; the radio telescope antenna of the Astronomical Observatory of the University of Tokyo with a total aperture diameter of 45 meters, constructed of 2.3 by 1.5 meters CFRP Al honeycomb panels; a thin solenoid for a superconducting magnet for the Tristan Project at the National Laboratory for High Energy Physics with a length of 5 meters, a

diameter of 3.8 meters, and a thickness of 27 mm fabricated by tape winding of CF prepreg tapes; and curtain walls for high rise buildings containing 3 vol % fiberglass or carbon fibers molded by a direct spray method. Additional applications include cooling water tanks, offshore structures, waterworks pipes, and naval and space satellite structures. While the market share of polymeric composite applications in Japan and the United States appears to be constant, the transportation applications have grown significantly between 1982 and 1986.

APPLICATIONS OF METAL MATRIX COMPOSITES

Metal composites in Japan can be divided into two categories, those with light matrices (such as Al and Mg) that must be used at low temperatures (below 400 °C) and those with more dense matrices that have potential high temperature (400 to 1,800 °C) capabilities. Al and Mg matrices have been reinforced with whiskers and fibers. The discontinuous Al and Mg composites are being developed primarily for commercial applications. These include automotive pistons (e.g., 200,000/month production levels) and connecting rods. There have also been a few aerospace components, missile fins, aircraft fuselage structures, etc. fabricated. However, structural components for military aircraft are likely in the future. Al and Mg matrices reinforced with continuous graphite or SiC fibers are being pursued for space application, e.g., satellite structural components and space antennas. Also, microwave devices (e.g., low CTE applications) and printed circuit boards are being considered. Goals for high temperature composites included capability for structural components for a 4,000-m/h aircraft. Jet engine materials capable of sustaining 1,800 °C operating temperatures are necessary. TiAl, NiAl, NbAl, and intermetallic composites are being considered in order to build engine components with these materials in 7 years. In the near term, the predominant opinion seems to be that there will be few commercial structural metal matrix composite markets within the next 5 years. With a longer term perspective, metal and ceramic matrix composites still hold the promise of higher specific property performance at elevated temperature and cannot be disregarded.

REFERENCES

1. T. Hayashi, *Sadhana*, II, Parts 3 & 4, 299-325 (December 1987).

2. T. Hayashi, *Proc. from 4th U.S.-Japan Conf. on Composite Materials*, Washington, D.C., June 1988 (Technomic Publications, in print).

3. T. Hayashi and Y. Minoda, private communications.

4. International Symposium on Basic Technologies for Future Industries - Materials Development and Technology Innovation - Poster Session, International Conference Center, Kobe, Japan, 22-25 March 1988.

5. Y. Minoda, "Composites '86: Recent advances in Japan and the United States," in *Proc. Japan-U.S. CCM-III*, edited by K. Kawata, S. Umekawa, and A. Kobayashi (Japan Society of Composite Materials, Tokyo, 1986).

6. T. Ishikawa, H. Ishikawa, and T. Teranishi, Nippon Carbon Co.

7. T. Yamamura et al., *J. Mat. Sci.* 23, 2589 (1988).

8. K. Okamura, M. Sato, T. Matsuzawa, T. Seguchi, and S. Kawanishi, "Silicon-based ceramic fibers," *Ceramic Eng. Sci. Proc.* 9(7-8), 909-918 (1988).

9. T. Matsuzawa, K. Okamura, and M. Sato, *Composites and Corrosion/Coating of Advanced Materials*, Proc. of International Meeting on Advanced Materials, vol 4, edited

by M. Doyama, S. Somiya, and R.P.H. Chang (Materials Research Society, Pittsburgh, Pa., 1989).

10. T. Isoda et al., Toa Nenryo Kogyo K.K., Central R&D.

11. E.A. Feest, M.J. Ball, A.R. Begg, and D.A. Biggs, Report from the OSTEM Mission Japan (Harwell Laboratory, October 1986).

12. Art Metal brochure, reprinted in Reference 11.

13. *Proc. from 4th U.S.-Japan Conf. on Composite Materials*, Washington, D.C., June 1988 (Technomic Publications, in print).

14. I.W. Hall, *Scripta Met.* 21, 1717-21 (1987).

15. K. Kuniya, H. Arakawa, and T. Namekawa, *Trans. Jap. Inst. of Metals* 28(3), 238-46 (1987).

16. Kobe Steel, Kobelco-ACM brochure, 1988.

17. CERAAI brochure, Mitsubishi Aluminum Corporation, undated.

18. T. Hattori and S. Sakai, *Mitsubishi Jukogikoo* 25(4), 1-5 (1987-8).

19. Y. Imai, H. Ichikawa, and T. Ishikawa, 20th Int. SAMPE Technical Conf., Minneapolis, Minnesota, 27-29 September 1988.

20. Nicalon brochure, Nippon Carbon Co., July 1987.

.... grains and the concomi-
tant compressive residual microstresses that
result in the matrix.

21. K. Akutagawa, H. Ohtsuki, J. Hasegawa, and M. Miyasaki, *SAE Technical Paper Series #870441*, Int. Congress and Exp. Detroit, Michigan, 23-27 February 1987.

22. N. Suzuki, K. Tanaka, and M. Yamanashi, UK Patent Application GB 2,162,104 A, 1986.

23. Showa Denko brochure, January 1987.

24. Sumitomo Chemical, "Sumitomo's alumina fiber products," brochure, 1988.

25. Tokawhisker - Silicon carbide whiskers, Tokai Carbon brochure, June 1988.

26. T. Kyono, E. Kuroda, A. Kitamura, T. Mori, and M. Taya, *J. of Eng. Materials and Techn.* 110, 89-95 (1988).

27. S.I. Towata, H. Ikuno, and S.I. Yamada, *Trans. Jap. Inst. of Metals* 29(4), 314-21 (1988).

28. S.I. Yamada, S.I. Towata, and H. Ikuno, in *Cast Reinforced Metal Composites*, edited by S.G. Fishman and A K. Dhingra, Proc. Conf. Chicago 1988 (ASM International, 1988), 109-14.

29. M. Kubo, A. Tanaka, and T. Kato, *J.S.A.E. Review* 9(3), 56-61 (1988).

30. T. Yamamura, Y. Waku, T. Yamamoto, M. Shibuya, M. Suzuki, T. Nishi, and T. Nagasawa, in *Looking Ahead for Materials and Processes*, Proc. 8th Int. Conf. of SAMPE, European Chapter, La Baule, France, 18-21 May 1987, 19-28.

31. H. Fukunaga, in *Cast Reinforced Metal Composites*, edited by S.G. Fishman and A.K. Dhingra, Proc Conf. Chicago 1988 (ASM International, 1988), 101-7.

32. S. Ochiai and K. Osamura, *Met. Trans.* 19A, 1491-1506 (1988).

33. M. Taya and T. Mori, *Acta Metall.* 35(1), 155-62 (1987).

34. T. Kobayashi, M. Yosino, H. Owanari, M. Niinomi and K. Yamamoto, in *Cast Reinforced Metal Composites*, edited by S.G. Fishman and A.K. Dhingra, Proc. Conf. Chicago 1988 (ASM International, 1988), 205-10.

35. T. Imai, Y. Nishida, M. Yamada, H. Matsubara, and I. Shirayanagi, *J. of Materials Sc. Letters* 6, 343-5 (1987).

36. A. Shindo and K. Honjo, in *Composite Interfaces*, Proc. First Int. Conf. on Composite Interfaces ICCI-1, Cleveland, Ohio, 1986, edited by H. Ishida and J.L. Koenig (North Holland, 1986) 93-100 and 101-7.

37. E. Yasuda, T. Akatsu, Y. Tanabe, Y. Katano, S. Kimura, and Y. Matsuo, "Development of SiC whisker reinforced alumina," in *Proc. of International Congress on Advanced Composites for New Systems and Technologies*, Weisbaden, West Germany, 19-22 September 1988.

38. K. Niihara, A. Nakahira, G. Sasaki, and M. Hirabayashi, "Development of strong Al_2O_3/SiC composites," in *Proc. of International Meeting on Advanced Materials* (Materials Research Society, Pittsburgh, Pa., 1988).

39. K. Matoba, K. Katayama, M. Kawanura, and T. Mizuno, "The development of second generation ceramic turbocharger rotor - Further improvements in reliability," in *Proc. of SAE Intern. Congress*, 29 February - 4 March 1988 (Society of Automotive Engineers Inc., Detroit, Michigan).

40. K. Niihara, T. Hirano, A. Nakahira, K. Ojima, K. Izaki, and T. Kawakami, "High temperature performance of Si_3N_4-SiC composites from fine amorphous Si-C-N powders," in *Structural Ceramics/Fracture Mechanics*, Proc. of International Meeting on Advanced Materials, vol 5, edited by Y. Hamano et al. (Materials Research Society, Pittsburgh, Pa., 1989).

41. S. Yamada, E. Yasuda, Y. Tanabe, and H. Yoneda, "In situ preparation of SiC whiskers in Si_3N_4 powder," *Ceramic Trans., Ceramic Powder Sci.*, edited by G. Messing, E. Fuller, and H. Hausner.

42. S. Yamada, S. Kimura, E. Yasuda, Y. Tanabe, and Y. Asami, "A discussion of the chemical mixing process for in situ preparation of silicon carbide whiskers in silicon nitride powder" *J. Mat. Res.* 3(3), 538-44 (1988).

43. N Tamari, M. Honda, and Y. Ebata, "Joining of silicon carbide ceramics with Si_3N_4-Y_2O_3-La_2O_3-MgO mixture," *Yogo-Kyokai-Shi* 94(10), 1087-91 (1986).

44. Y. Akimune, Y. Katano, and Y. Shichi, "Microstructure and mechanical properties of SiC-whisker/Y-TZP composites," *Sintering 1987*, Tokyo.

45. Y. Akimune, Y. Katano, and Y. Shichi, "Mechanical properties and microstructure of air-annealed SiC-whisker/Y-TZP composites," *Advan. Cer. Mater.* 3(3), 138-42 (1988).

46. K. Terao, Y. Miyamoto, and M. Koizumi, "Characteristics of ZrO_2-dispersed Si_3N_4 without additives fabricated by hot isostatic pressing," *J. Amer. Soc.* 71, C-167-9 (1988).

47. S. Masuda and T. Itoh, Conference Record of the 1987 IEEE Industry Applications Society Annual Meeting, Part II.

48. K. Nakano, A. Kondo, and A. Kamiya, "Structure and mechanical properties of SiC reinforced spinel ceramics," in *Composites and Corrosion/Coating of Advanced Materials*, Proc. of International Meeting on Advanced Materials, vol 4, edited by M. Doyama, S. Somiya, and R.P.H. Chang (Materials Research Society, Pittsburgh, Pa., 1989).

49. T. Watanabe and K. Shobu, "Effects of TiC additive on the mechanical properties of TiB_2-TaB_2-CoO-TiC materials obtained from fine powder," *Yogo-Kyokai-Shi* 96(7), 778-82 (1988).

50. K. Shobu and T. Watanabe, "Sintering and oxidation of Ti(C,N)-TiB_2-$MoSi_2$ composites," *Yogo-Kyokai-Shi* 95(1), 991-99 (1987).

51. E. Fitzer and R. Gadow, "Fiber reinforced silicon carbide," *Am. Cer. Soc. Bull.* 65(2), 326-35 (1986).

52. K. Nakano, A. Kamiya, T. Suganuma, I. Maki, and A. Kanno, "Fabrication of fiber reinforced silicon carbide composites," *Proc. of Sintering*, Japan (1987).

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